



TRIBOLOGY INTERNATIONAL CONFERENCE



# SICT 2022 / PLASMA TECH 2022 / TRIBOLOGY 2022

## HYBRID JOINT CONFERENCES

27-29 Avril, 2022 - Barcelona, Spain

## Book of Abstracts

Organizer



**SETCOR**  
Conferences & Exhibitions

# SICT 2022 / PlasmaTech 2022 / Tribology 2022 Joint Hybrid Conferences Program

27 - 29 April 2022 | Barcelona, Spain

27 April 2022		
08:00 - 12:00	Onsite Participant registration.	
08:30 - 10:00	Welcoming Coffee	
SICT 2022 / Plasma Tech 2022 / Tribology 2022 Joint Plenary Onsite session		
Onsite Conference Room Glorias A		
Session's Chairs: Prof. Holger Kersten, University Kiel, Germany Prof. Alicia de Andrés, Materials Science Institute of Madrid (CSIC), Spain Prof. Alessandro Ruggiero, University of Salerno, Italy		
10:00 - 10:30	Continuous to Nanoflake MoS2 Single-Layers: Characterization and Sensing Applications using Optical Spectroscopies. A. de Andrés, S. Cortijo-Campos, L. Álvarez-Fraga and C. Prieto	Prof. Alicia de Andrés, Materials Science Institute of Madrid (CSIC), Spain
10:30 - 11:00	Post-Thermal Spray Coating Surface Modification and Potential Industrial Applications A. Amanov and Y.S. Pyun	Prof. Auezhan Amanov, Sun Moon University, Rep. of Korea
11:00 - 11:30	The Energy Balance of Nano- and Microparticles during Plasma Processing H. Kersten	Prof. Holger Kersten, University Kiel, Germany
11:30 - 12:00	Graphene nanowalls grown on stainless steel by inductively coupled plasma chemical vapor deposition E. Bertran-Serra, S. Chaitoglou, R. Amade Rovira, A. Mushegyan-Avetishyan, I. Alshaikh, L. F. Pantoja-Suarez, J. L. Andújar Bella, A. Perez-del-Pino and E. Georgy	Prof. Enric Bertran-Serra, Barcelona University, Spain
12:00 - 14:00	Lunch Break	
SICT 2022 Session I: Surfaces and Coatings processing / Characterization / Properties Multifunctional composite and hybrid coatings		
Onsite Conference Room Glorias A		
Session's Chairs: Prof. Alicia de Andrés, Materials Science Institute of Madrid (CSIC), Spain Prof. Auezhan Amanov, Sun Moon University, Rep. of Korea Dr. Himanshu H Mishra, King Abdullah University of Science & Technology, KSA		
14:00 - 14:30	A comparative study of Ir(dmpq)2(acac) doped CBP, mCP, TAPC and TCTA for phosphorescence OLEDs D. Tselekidou, L. Panagiotidis, K. Papadopoulos, V. Kyriazopoulos and M. Gioti	Prof. Maria Gioti, Aristotle University of Thessaloniki, Greece
14:30 - 14:45	Synthesis of freestanding porous alumina and metallic nanowires S. Ionescu, C. Gheorghiu, D. Popa and V. Leca	Ms. Stefania-Cristina Ionescu, National Institute for Physics and Nuclear Engineering, Romania
14:45 - 15:00	Tunable thermal and photochemical crosslinking of CHic-able diazo-groups containing polyers D. Rusitov and J. Rühle	Mr. Dennis Rusitov, University of Freiburg, Freiburg, Germany
15:00 - 15:15	Dual-phase CuZr thin film metallic glasses (TFMGs) deposited by PVD magnetron sputtering: bias voltage and thickness dependency of structure, hardness, and wear resistance A. Bagherpour, M-S. Colla b, A. Orekhov, T. Pardoen and S. Lucas	Mr. Alireza Bagherpour, The University of Namur, Belgium
15:15 - 15:30	Post Processed Ceramic Based Coatings on Steels using Thermal Spray A. Pattnayak, A. Gupta, N.V Abhijith, D. Kumar, J. Jain and V. Chaudhry	Mr. Abhijit Pattnayak, Indian Institute of Technology Delhi, India



15:30 - 15:45	Effects of 18 Ni (300) maraging steel coating by laser deposition on LPBF AISI 316L component <b>V. Errico</b> , P. Posa, A. Angelastro, M. Mazzarisi, A. Fusco and S.L. Campanelli	<b>Mr. Vito Errico</b> , The Polytechnic University of Bari, <b>Italy</b>
15:45 - 16:00	Properties of coatings and SPS sinters made of tungsten diboride alloyed with Ti, Cr, Mo, Re and Zr <b>R. Psiuk</b> , M. Wiśniewska, D. Garbiec and T. Mościcki	<b>Mr. Rafal Psiuk</b> , Institute of Fundamental Technological Research, <b>Poland</b>
16:00 - 16:30	Coffee Break	
Session's Chairs: <b>Prof. Alicia de Andrés, Materials Science Institute of Madrid (CSIC), Spain</b> <b>Prof. Auezhan Amanov, Sun Moon University, Rep. of Korea</b> <b>Dr. Ramon Escobar Galindo, University of Seville, Spain</b>		
16:30 - 16:45	The Influence of Ambient Cure Chemistry and Stoichiometry on the Surface Properties of Epoxy Coatings for Industrial Application <b>C. Bannister</b> , A. Guy and R. Thompson	<b>Mr. Callum Bannister</b> , Durham University, <b>UK</b>
16:45 - 17:00	Study of bioinspired super-hydrophobic polymer textured surfaces to design fluorine-free high repellency textiles <b>Q. Legrand</b> , S. Benayoun and S. Valette	<b>Dr. Quentin Legrand</b> , Ecole Centrale de Lyon, <b>France</b>
17:00 - 17:15	Development and investigation of textile materials with Conductive Coatings intended for microwave shielding and absorption <b>V. Rubeziene</b> , A. Sankauskaitė, S. Varnaitė-Žuravliova and A. Abraitienė	<b>Dr. Vitalija Rubeziene</b> , Center for Physical Sciences and Technology- Kaunas, <b>Lithuania</b>
17:15 - 17:30	Oxidation Kinetics of Inconel 625® under CO2 <b>B. Contri</b> , S. Valette, M. Soustre and P. Lefort	<b>Mr Boris Contri</b> , University of Limoges, <b>France</b>
17:30 - 17:45	Evaluation and improvement of adhesion between polymeric nano-fibers and metallic substrates <b>T. Schneiders</b> , F. Ahrens and T. Gries	<b>Mr. Thomas Schneiders</b> , RWTH Aachen University, <b>Germany</b>
17:45 - 18:00	Effect of the detonation spraying technological parameter on the phase composition and properties of Cr3C2- NiCr coatings <b>N.M.Magazov</b> , B.K. Rakhadilov D.N. Kakimzhanov and L.G. Zhurerova	<b>Mr. Nurtoleu Magazov</b> , Daulet Serikbayev East Kazakhstan technical university, <b>Kazakhstan</b>
18:00 - 18:15	Hybrid nanostructures for (photo)electrochemical water splitting <b>S. Kment</b> , A. Naldoni and R. Zboril	<b>Dr. Stepan Kment</b> , Palacky University Olomouc, <b>Czech Republic</b>
18:15 - 18:30	The effect of cold plasma and fs-laser generated plasma surface treatment on the wettability of high strength steel and the strength of the overlap bonded joint <b>Z. Weltsch</b> and M. Berczeli	<b>Dr. Zoltan Weltsch</b> , John von Neumann University, <b>Hungary</b>
18:30 - 18:45	Influence of cold plasma surface treatment of HIPS and PA6 based nanocomposite polymers on wetting properties and 2K epoxy and 2K hybrid adhesive joints <b>M. Berczeli</b> and Z. Weltsch	<b>Mr. Miklos Berczeli</b> , John von Neumann University, <b>Hungary</b>

27 April 2022

**Plasma Tech Session I**  
**Plasma fundamentals / Modelling / Atomic and Molecular Processes**

**Onsite Conference Room Glorias B**

**Session's Chairs:**

**Prof. Enric Bertran-Serra, Barcelona University, Spain**  
**Dr. Thomas Danny Michl, University of Applied Sciences and Arts Northwestern Switzerland, Switzerland**  
**Prof. Vasco Guerra, Technical University of Lisbon, Portugal**

<b>14:00 - 14:30</b>	Controlling Atmospheric Pressure Plasma Interactions with Solids and Liquids Kseniia Konina, Mackenzie Meyer, Sanjana Kerketta, Astrid Raisanen, Jordyn Polito and <b>Mark J. Kushner</b>	<b>Prof. Mark J. Kushner</b> , University of Michigan, <b>USA</b>
<b>14:30 - 14:45</b>	Plasma spraying related modelling and experimental studies <b>M. Baeva</b> , T. Zhu, H. Testrich, R. Methling and R. Foest	<b>Dr. Margarita Baeva</b> , Leibniz Institute for Plasma Science and Technology, <b>Germany</b>
<b>14:45 - 15:00</b>	Non-Thermal Plasma Sources Based on Cometary and Point-to-Ring Discharges <b>E. Lokajová</b> , J. Khun, A. Machková, P. Kašparová, M. Klenivskiy, E. Vaňková, P. Galář, J. Julák and V. Scholtz	<b>Mrs. Eliska Lokajova</b> , University of Chemistry and Technology- Prague, <b>Czech Republic</b>
<b>15:00 - 15:15</b>	A modelling investigation of the mechanisms underlying the O/O <sub>2</sub> removal in the afterglow of a CO <sub>2</sub> plasma with a carbon bed <b>O. Biondo</b> , F. Girard-Sahun, G. Trenchev, G. van Rooij and A. Bogaerts	<b>Mr. Omar Biondo</b> , The University of Antwerp, <b>The Netherlands</b>
<b>15:15 - 15:30</b>	Behaviour of Electric Arc in Plasma-Chemical Reactor during Hazardous Waste Processing <b>V. Grigaitienė</b> , D. Gimžauskaitė, V. Valinčius, R. Kėželis, R. Uscila and Ž. Kavaliauskas	<b>Dr. Viktorija Grigaitiene</b> , Lithuanian Energy Institute, <b>Lithuania</b>
<b>15:30 - 15:45</b>	Global plasma modelling of PECVD growth of SiO <sub>2</sub> films for optical applications <b>K. Tomankova</b> , N. Rivolta and A. Obrusnik	<b>Ms. Kristina Tomankova</b> , PlasmaSolve company, <b>Czech Republic</b>
<b>15:45 - 16:00</b>	Characterization of non-thermal coronal plasma discharges for different high voltage electrode configurations in the atmosphere using air. S.O. Babalola, <b>V. Tshigo</b> , M.O. Daramola and S.A. Iwarere	<b>Mr Victor Tshigo</b> , University of Pretoria, <b>South Africa</b>
<b>16:00 - 16:15</b>	Investigations of the sheath in a dual-frequency capacitively coupled rf discharge by optically trapped microparticles <b>J. Schleitzer</b> , V. Schneider and H. Kersten	<b>Ms. Jessica Schleitzer</b> , Christian-Albrechts Univ., <b>Germany</b>

27 April 2022

**SICT 2022 / Tribology 2022**  
**ReSISTant EU Project Workshop: Drag Reducing Surfaces (Riblets) - Simulations, high resistant nano-coatings development, applications, and new manufacturing processes for future worldwide applications**

**Onsite Conference Room Glorias C**

08:30 - 10:00

Welcoming Coffee

**Workshop Chairs:**  
**Dr. Andreas Flanschger, Bionic surface technologies GmbH, Austria**

10:00 - 10:15	Overview Project ReSISTant <b>A. Flanschger</b>	<b>Dr. Andreas Flanschger</b> , Bionic surface technologies GmbH, Austria
10:15 - 10:45	Industrial Compressors: Riblet Simulation Results <b>L.G. de Albeniz Martinez</b>	<b>Mr. Lucas Garcia de Albeniz Martinez</b> , Bionic surface technologies GmbH, Austria
10:45 - 11:15	Industrial Compressors: Applications and Test results <b>M. Meyer</b>	<b>Mr. Michael Meyer</b> , MAN Energy Solutions Schweiz AG, Switzerland
11:15 - 11:45	Riblet application technologies I: Coatings for harsh conditions <b>A. Brinkmann</b>	<b>Mr. Andreas Brinkmann</b> , Fraunhofer IFAM, Germany
11:45 - 12:15	Aircraft Turbines: Riblet Simulation Results <b>L.G. de Albeniz Martinez</b>	<b>Mr. Lucas Garcia de Albeniz Martinez</b> , Bionic surface technologies GmbH, Austria

12:00 - 14:00

Lunch Break

14:00 - 14:15	Project ReSiSTant and beyond – new Riblet applications <b>Flanschger</b>	<b>Dr. Andreas Flanschger</b> , Bionic surface technologies GmbH, Austria
14:15 - 14:45	Riblet application technologies II: Riblet Film and beyond <b>C. Baum</b>	<b>Dr. Christoph Baum</b> , Polyscale GmbH & Co. KG, Germany
14:45 - 15:15	Riblet Application Technologies III: Laser <b>Y. Shibazaki</b>	<b>Mr. Yuichi Shibazaki</b> , NIKON Corporation, Japan
15:15 - 15:45	Riblet Application Technologies IV: Direct Contactless Microfabrication <b>H. Bilinsky</b>	<b>Mr. Henry Bilinsky</b> , MICROTAU PTY LTD, Australia

16:00 - 16:30

Coffee Break

16:30 - 17:00	Riblet Material Development: Antifouling Riblet coatings <b>A. Brinkmann</b>	<b>Mr. Andreas Brinkmann</b> , Fraunhofer IFAM, Germany
17:00 - 17:30	Surface Characterization: Roughness direct simulation <b>L.G. de Albeniz Martinez</b>	<b>Mr. Lucas Garcia de Albeniz Martinez</b> , Bionic surface technologies GmbH, Austria
17:30 - 18:00	Riblet Material Development: Riblet Defects and their impact <b>C. Feichtinger</b>	<b>Mr. Christoph Feichtinger</b> , Bionic surface technologies GmbH, Austria

<b>28 April 2022</b>		
<b>Plasma Tech Session II: Plasma Processing / materials interactions / coatings</b>		
<b>Onsite Conference Room Glorias A</b>		
<b>Session's Chairs:</b> <b>Prof. Mark J. Kushner, University of Michigan, USA</b> <b>Prof. Vasco Guerra, Technical University of Lisbon, Portugal</b>		
<b>09:00 - 09:30</b>	Selectively changing key surface properties via atmospheric gliding arc plasma deposition <b>T. Danny Michl</b> , A. Goel and S. Neuhaus	<b>Dr. Thomas Danny Michl</b> , University of Applied Sciences and Arts Northwestern Switzerland, <b>Switzerland</b>
<b>09:30 - 09:45</b>	On the formation of carbon nanoparticles in expanding laser-induced plasma <b>A. Kaczmarek</b> and J. Hoffman	<b>Ms. Agata Kaczmarek</b> , Institute of Fundamental Technological Research Polish Academy of Sciences, <b>Poland</b>
<b>09:45 - 10:00</b>	Carbon nanostructure production from ethanol by cold plasma <b>A. Jurov</b> , J. Zavašnik and U. Cvelbar	<b>Dr. Andrea Jurov</b> , University of Zagreb, <b>Croatia</b>
<b>10:00 - 10:15</b>	Hydrophobic and Amphiphobic Postmodification of Mesoporous Aerogels via Cold Plasma Coating <b>B. Schroeter</b> , I. Jung, P. Gurikov and I. Smirnova	<b>Dr. Baldur Schroeter</b> , Hamburg University of Technology, <b>Germany</b>
<b>10:15 - 10:30</b>	Plasma Activated Liquids: a Method for Efficient Surface Modification of Semiconductor Nanostructures <b>P. Galář</b> , F. Matějka, J. Khun and K. Kůsová	<b>Dr. Pavel Galář</b> , Institute of Physics of the Czech Academy of Sciences, <b>Czech Republic</b>
<b>10:30 - 11:00</b>	<b>Coffee Break</b>	
<b>11:00 - 11:15</b>	UV-LED, UV-laser and Corona discharge treatments for polypropylene surface functionalization and optimization of PP-Fiber Reinforced Concrete <b>B. Malchiodi</b> , P. Pozzi and C. Siligardi	<b>Mrs Beatrice Malchiodi</b> , University of Modena and Reggio Emilia, <b>Italy</b>
<b>11:15 - 11:30</b>	Fast Switch From Hydrophilic to Hydrophobic Surface of Cellulose Film by Low-Temperature Plasma Treatment <b>A. Oberlintner</b> , V. Shvalya, B. Likozar and U. Novak	<b>Ms Ana Oberlintner</b> , National Institute of Chemistry, <b>Slovenia</b>
<b>11:30 - 11:45</b>	Recent progress in the electrical management of the plasma electrolytic oxidation process <b>J. Martin</b> , V. Ntomprougkidis, C. Tousch, A. Maizeray, G. Marcos, T. Czerwicz, T. Belmonte and G. Henrion	<b>Dr Julien Martin</b> , University of Lorraine, <b>France</b>
<b>11:45 - 12:00</b>	Promoting resource preservation by PECVD barrier coatings for refillable PET bottles <b>P. Alizadeh</b> and R. Dahlmann	<b>Dr. Philipp Alizadeh</b> , RWTH Aachen University, <b>Germany</b>
<b>12:00 - 12:15</b>	Cathodic plasma electrolytic deposition of an aluminium oxide based hydrogen permeation barrier <b>M. Wetegrove</b> , M. Rohloff, U. Lindemann, A. Quade and A. Kruth	<b>Dr. Marcel Wetegrove</b> , Leibniz Institute for Plasma Science and Technology, <b>Germany</b>
<b>12:00 - 14:00</b>	<b>Lunch Break</b>	

**Plasma Tech Session III:  
Plasma application for biology, medicine, and agriculture**

**Onsite Conference Room Glorias A**

**Session's Chairs:**  
**Prof. Lia-Mara Ditu, University of Bucharest, Romania**  
**Prof. Cristina Canal, The Polytechnic University of Catalonia, Spain**  
**Dr. Ita Junkar, Jožef Stefan Institute, Slovenia**

<b>14:00 - 14:30</b>	Challenges in Plasma-Conditioned Liquids and Hydrogels for Cancer Treatment C. Labay, F. Tampieri, A. Espona, X. Solé-Martí, M. Mateu-Sanz, J. Tornin and <b>C. Canal</b>	<b>Prof. Cristina Canal</b> , The Polytechnic University of Catalonia, <b>Spain</b>
<b>14:30 - 15:00</b>	Multifunctional medical materials of the future by plasma surface modification <b>I. Junkar</b> and <b>M. Benčina</b>	<b>Dr. Ita Junkar</b> , Jožef Stefan Institute, <b>Slovenia</b>
<b>15:00 - 15:15</b>	Improved biocompatibility of hydrothermally and plasma-treated titanium <b>M. Benčina</b> , N. Rawat, P. Starič, K. Lakota, S. Sodin-Šemrl, A. Iglič and I. Junkar	<b>Dr. Metka Bencina</b> , Jožef Stefan Institute, <b>Slovenia</b>
<b>15:15 - 15:30</b>	PECVD coatings as migration barriers for the use of post-consumer recyclates in food contact <b>L. Kleines</b> and R. Dahlmann	<b>Mrs. Lara Kleines</b> , RWTH Aachen University, <b>Germany</b>
<b>15:30 - 15:45</b>	Utilization of Plasma in Medicine: Inactivation of Acanthamoebas, Dermatophytes, and Staphylococci <b>T. Měřínská</b> , E. Lokajová, J. Julák, E. Nohýnková, J. Khun, V. Scholtz and P. Petráš	<b>Ms. Tereza Merinska</b> , University of Chemistry and Technology- Prague, <b>Czech Republic</b>
<b>15:45 - 16:00</b>	Understanding the molecular mechanisms of non-thermal plasma treatments on Arabidopsis thaliana seeds <b>A. Waskow</b> , A. Guihur, A.A. Howling and I. Furno	<b>Ms. Alexandra Waskow</b> , EPFL university, <b>Switzerland</b>
<b>16:00 - 16:15</b>	Inactivation of Critically Ranked Carbapenem Resistant Bacteria and Genes Using Cold Atmospheric Plasma Technology <b>T.B.M. Mosaka</b> , M.O. Daramola, C. Tizaoui and S.A. Iwarere	<b>Ms. Thabang B.M. Mosaka</b> , University of Pretoria, <b>South Africa</b>
<b>16:00 - 16:30</b>	<b>Coffee Break</b>	

28 April 2022

**SICT 2022 / Tribology 2022 Joint Session I:  
Coatings and Surfaces Corrosion / tribological properties / adhesion and Adhesives**

**Onsite Conference Room Glorias B**

**Session's Chairs:**

**Dr. Himanshu H Mishra, King Abdullah University of Science & Technology, KSA**

08:30 - 09:00	Corrosion Protection Potential of Hard Nitride Based Coatings Produced with PVD techniques <b>M. Ürgen</b> and B. Avci	<b>Prof. Mustafa Urgen</b> , Istanbul Technical University, Turkey
09:00 - 09:15	Anticorrosive PEO coatings on metallic cast heat enhancers for thermal energy storage <b>N. Ražny</b> , A. Dmitruk and K. Naplocha	<b>Ms. Natalia Ražny</b> , Wrocław University of Science and Technology, Poland
09:15 - 09:30	Self-healing core-shell nanofibres for corrosion protective coatings <b>N. C. M. Spera</b> and J. P. S. Sousa	<b>Dr. Natalia Spera</b> , Iberian Nanotechnology Laboratory, Portugal
09:30 - 09:45	Epoxy Novolac Coating Interactions at High Pressure High Temperature Conditions <b>N. Rajagopalan</b> , C.E. Weinell, K. Dam-Johansen and S. Kiil	<b>Dr. Narayanan Rajagopalan</b> , Technical University of Denmark, Denmark
09:45 - 10:00	Mechanisms of corrosion protection of steel using industrial water-borne zinc rich sol-gel coatings <b>A. Boidot</b> , C. Jama, J-B. Vogt and F. Gheno	<b>Mr. Arthur Boidot</b> , Ecole Centrale Lille, France
10:00 - 10:15	Pull-off strength of polyurethane coatings with waste rubber fillers on aluminum substrate after aged and thermal shock processes <b>P. Mayer</b> , M. Ferraris and S. Perero	<b>Dr. Paulina Mayer</b> , Wrocław University of Science and Technology, Poland
10:00 - 10:30	Tungsten carbide surfaces: challenging PVD coatings by additive manufacturing <b>F. Koehn</b> , W. Schulz and J. Albrecht	<b>Mr. Florian Koehn</b> , Aalen University, Germany

10:30 - 11:00

Coffee Break

**Session's Chairs:**

**Prof. Mustafa Urgen, Istanbul Technical University, Turkey**

**Dr. Himanshu H Mishra, King Abdullah University of Science & Technology, KSA**

11:00 - 11:15	Adhesive and wear behavior of a duplex coating of a nitrided layer plus diamond-like carbon coating. <b>I. Gómez</b> , A. Claver, J.A. Santiago, J. Fernandez, Bracerias and J.A. Garcia	<b>Prof. Iñigo Gómez Alonso</b> , Public University of Navarre, Spain
11:15 - 11:30	Wear behavior of single stage HVOF sprayed TiNbMoMnFe high entropy alloys coating <b>N.V. Abhijith</b> , A. Pattnayak, A. Gupta and D. Kumar	<b>Mr. N.V. Abhijith</b> , Indian Institute of Technology Delhi, India
11:30 - 11:45	Tribological Characterization of Ti-based Magnetron Sputtered Thin Films: A small-scale in situ study <b>A. Sayilan</b> , J. Ferreira, C. Goudin, C. Lopes, J. Borges, F. Vaz, N. Mary, S. Descartes and P. Steyer	<b>Ms. Aslihan Sayilan</b> , MATEIS, Univ. Lyon/ INSA-Lyon, France
11:45 - 12:00	Controlling the tribological properties of hard coatings by substrate engineering <b>W. Schulz</b> , F. Köhn and J. Albrecht	<b>Mr. Wadim Schulz</b> , Aalen University, Germany
12:00 - 12:15	Effect of Substrate (WC-Co) Surface Roughness on the Tribological Properties of DLC Coatings Deposited by HiPIMS <b>S.A. Khan</b> , J. Oliveira, N. Emami and A. Ramalho	<b>Mr. Sharjeel Ahmed Khan</b> , University of Coimbra, Portugal
12:15 - 12:30	Antiadhesive coating in aqueous phase to prevent colonization by microorganisms <b>M. Champion</b> , I. Linossier, K. Rehel, C. Hellio, X. Moppert and F. Faÿ	<b>Ms. Marie Champion</b> , University of South Brittany, France

12:00 - 14:00

Lunch Break



28 April 2022

**SICT 2022 / Tribology 2022 joint Session II:  
Physics or chemistry of tribo-surfaces/ Nanotribology (Part I)**

**Onsite Conference Room Glorias C**

**Session's Chairs:**

**Dr. Chen Xiao, Advanced Research Center for Nanolithography, The Netherlands**

**Dr. Alberto Rota, University of Modena and Reggio Emilia, Italy**

**Prof. Shih-Chieh Lin, National Tsing Hua University, Taiwan**

<b>08:30 - 09:00</b>	Adhesion-aided friction of viscoelastic materials <b>G. Carbone</b> , C. Mandriota and N. Menga	<b>Prof. Giuseppe Carbone</b> , Polytechnic University of Bari, Italy
<b>09:00 - 09:30</b>	Compositional and topographical tailoring of tungsten carbide surfaces to control friction and wear under dry conditions <b>J. Albrecht</b> , W. Schulz and F. Köhn	<b>Prof. Joachim Albrecht</b> , Aalen University, Germany
<b>09:30 - 10:00</b>	Experimental and modelling-based upscaling of new material concept for journal bearings <b>H. Ronkainen</b> , J. Tervo, M. Savolainen, J. Virtanen and Sami Majaniemi	<b>Prof. Helena Ronkainen</b> , VTT Technical Research Centre, Finland
<b>10:00 - 10:15</b>	Challenges to Testing - Rolling Contact Fatigue on Modern High Strength Steels <b>V. Heino</b> , R. Parikka, M. Lindroos and H. Ronkainen	<b>Dr. Vuokko Heino</b> , VTT Technical Research Centre of Finland Ltd, Finland
<b>10:15 - 10:30</b>	The Dynamics of Capillary Bridge Formation in the Non-Contact Regime <b>F. Cassin</b> and B. Weber	<b>Dr. Felix Cassin</b> , Advanced Research Center for Nanolithography, The Netherlands

**10:30 - 11:00**

**Coffee Break**

**Session's Chairs:**

**Prof. Giuseppe Carbone, Polytechnic University of Bari, Italy**

**Prof. Helena Ronkainen, VTT Technical Research Centre, Finland**

<b>11:00 - 11:30</b>	Recent in-silico models for wear calculation in total hip replacements <b>A. Ruggiero</b>	<b>Prof. Alessandro Ruggiero</b> , University of Salerno, Italy
<b>11:30 - 11:45</b>	Wear Particle Dynamics Drives the Difference between Repeated and Non-Repeated Reciprocating Sliding F-C. Hsia, F. Elam, D. Bonn, B. Weber and <b>S. Franklin</b>	<b>Dr. Chen Xiao</b> , Advanced Research Center for Nanolithography, The Netherlands
<b>11:45 - 12:00</b>	Fine Defect Engineering of Graphene Friction <b>A. Zambudio</b> , E. Gnecco, J. Colchero, R. Pérez, J. Gómez-Herrero and C. Gómez-Navarro	<b>Mr. Aitor Zambudio Sepúlveda</b> , The Autonomous University of Madrid, Spain
<b>12:00 - 12:15</b>	Investigation of experimental production parameters effects on the wear behaviour of copper-tungsten disulfide composite <b>M. Freschi</b> , L. Dragoni, M. Mariani, O. Haiko, J. Kömi, N. Lecis and G. Dotelli	<b>Mr. Marco Freschi</b> , Polytechnic university of Milan, Italy
<b>12:15 - 12:30</b>	Fabrication of generated rough surface topographies through additive manufacture for replication and study in various polymer materials <b>J. Perris</b> , C. Kumar, Y. Xu, N. Gadegaard and D. Mulvihill	<b>Mr. Jack Perris</b> , University of Glasgow, UK

**12:00 - 14:00**

**Lunch Break**

## SICT 2022 / Tribology 2022: i-Tribomat Workshop

**Session's Chairs:**  
**Dr Xavier Borrás, AC2T Research, Austria**

<b>14:00 - 14:45</b>	AC2T – The European Tribology Centre <b>F. Pirker</b>	<b>Dr. Franz Pirker</b> , AC2T research GmbH, <b>Austria</b>
<b>14:45 - 15:00</b>	On the Challenges in Achieving Comparable and Reproducible Tribological Model Tests <b>M. Kröll</b>	<b>Mr. Mirco Kröll</b> , Federal Institute of Materials Research and Testing (BAM), <b>Germany</b>
<b>15:00 - 15:15</b>	Seamless Data Flow <b>J. Benedicto</b> and A. Garcia	<b>Mr. Jesus Benedicto</b> , ATOS, <b>Spain</b>
<b>15:15 - 15:30</b>	The Tribology Database <b>D. Dykeman</b>	<b>Dr. Donna Dykeman</b> , Ansys UK Ltd, <b>UK</b>
<b>15:30 - 15:45</b>	The Art of Downscaling & Upscaling <b>U. Cihak-Bayr</b>	<b>Dr. Ulrike Cihak-Bayr</b> , AC2T research GmbH, <b>Austria</b>
<b>15:45 - 16:00</b>	Success Story of TRYGONAL IBERIA <b>F. Pagano</b>	<b>Dr. Francesco Pagano</b> , Tekniker Foundation, <b>Spain</b>
<b>16:00 - 16:30</b>	<b>Coffee Break</b>	
<b>16:30 - 16:45</b>	Game Changing in Material Development through Lab-to-Field Approaches <b>I. Minami</b>	<b>Prof. Ichiro Minami</b> , Luleå University of Technology, <b>Sweden</b>

## SICT 2022 / Tribology 2022 joint Session II: Physics or chemistry of tribo-surfaces / Nanotribology (Part II)

### Onsite Conference Room Glorias C

**Session's Chairs:**  
**Prof. Joachim Albrecht, Aalen University, Germany**  
**Prof. Mitjan Kalin, University of Ljubljana, Slovenia**

<b>17:00 - 17:15</b>	Effect of humidity on capillary adhesion and friction at silicon-on-silicon multi-asperity interfaces <b>L. Peng</b> , F.-C. Hsia, S. Woutersen, B. Weber and, D. Bonn	<b>Mr. Liang Peng</b> , University of Amsterdam, <b>The Netherlands</b>
<b>17:15 - 17:30</b>	Frictional properties and nano-mechanical analysis of surface-attached hydrogels <b>R. Maraula</b> and J. Rühe	<b>Mr. Renato Maraula</b> , Albert Ludwig University, Freiburg, <b>Germany</b>
<b>17:30 - 17:45</b>	Ti3C2Tx Nano-Sheets: Substrate-Dependent Tribo-Chemical Reactions <b>A. Rota</b> , N. Bellina and A. Rosenkranz	<b>Dr. Alberto Rota</b> , University of Modena and Reggio Emilia, <b>Italy</b>
<b>17:45 - 18:00</b>	A Novel Restrictor Design for Hydrostatic Bearing <b>S. C. Lin</b> , Y. H. Lo, Y. H. Lin, W. T. Tung and T. H. Lai	<b>Prof. Shih-Chieh Lin</b> , National Tsing Hua University, <b>Taiwan</b>
<b>18:00 - 18:15</b>	Skin friction: mechanical and tribological characterization of different papers used in everyday life <b>L.M. Vilhena</b> and A. Ramalho	<b>Dr. Luis Vilhena</b> , University of Coimbra, <b>Portugal</b>



29 April 2022

## SICT Session II: Coatings for Energy and Environmental Applications

### Onsite Conference Room Glorias B

#### Session's Chairs:

**Prof. Iñigo Gómez Alonso, Public University of Navarre, Spain**

**Prof. Maria Gioti, Aristotle University of Thessaloniki, Greece**

08:30 - 09:00	Optical design, microstructural characterization and high-temperature in-air stability study of solar selective coatings based on aluminium- (titanium, chromium) oxynitride multilayers <b>R. Escobar Galindo</b> , I. Heras, E. Guillén, F. Munnik, I. Azkona, A. Caro, T.C. Rojas, J.C. Sánchez-López and M. Krause	<b>Dr. Ramon Escobar Galindo</b> , University of Seville, <b>Spain</b>
09:00 - 09:30	Nature-Inspired Coating Technologies for Global Food-Water Security <b>H. Mishra</b>	<b>Dr. Himanshu H Mishra</b> , King Abdullah University of Science & Technology, <b>KSA</b>
09:30 - 09:45	Synthesis of Graphene-suspensions by plasma-in-liquid process for thin film coatings of polymer-membranes for fuel cell application. <b>C. Rojas</b> , T. Schulz, F. Käufer, P. Quarz, V. Bravo, J. Kapp, V. Lukasek, T. Sommer, P. Scharfer, C. Scheu, J. Wartmann, W. Schabel and A. Kruth	<b>Mrs. Camila A. Rojas Nuñez</b> , Leibniz Institute for Plasma Science and Technology, <b>Germany</b>
09:45 - 10:00	Impact of Crosslinker Chemistry and Concentration on Reinforcement of GO Composites for Water Filtration Applications <b>P. Kaur</b> , R.L. Thompson, L.R. Hutchings, M.U. Chaudhary and T. Pugh	<b>Ms. Purneema Kaur</b> , Durham University, <b>UK</b>

10:00 - 10:30

Coffee Break

## Plasma Tech Session IV: Plasma application in Energy and environment

### Onsite Conference Room Glorias B

#### Session's Chairs:

**Prof. Holger Kersten, University Kiel, Germany**

10:30 - 11:00	Validation of complex plasma chemistries: CO <sub>2</sub> as a case study <b>V. Guerra</b> , T. Silva, C. Fromentin, T. C. Dias, A. S. Morillo-Candas and O. Guaitella	<b>Prof. Vasco Guerra</b> , Technical University of Lisbon, <b>Portugal</b>
11:00 - 11:15	Plasma-ammonia formation integrating high-temperature hydrogen plasma formation and biomass gasification <b>N.V.D. Long</b> , K. van 't Veer, J. Osorio Tejada, N. N. Tran, L. Fulcheri, B. Patil, V. Hesse, A. Bogaerts	<b>Dr. Long Nguyen</b> , University of Warwick, <b>UK</b>
11:15 - 11:30	Plasma-catalytic coupling in a nanosecond pulsed discharge plasma for CO <sub>2</sub> recycling <b>M. Faedda</b> , B. Samojeden, L.M. Martini, M. Motak and P. Tosi	<b>Ms. Marzia Faedda</b> , AGH University of Science and Technology, <b>Poland</b>
11:30 - 11:45	Effect of morphology of nanostructured CeO <sub>2</sub> supports on plasma-assisted CO <sub>2</sub> methanation <b>B. Musig</b> , M.E. Gálvez and M.V. Navarro	<b>Ms. Beatrice Musig</b> , Instituto de Carboquímica-Zaragoza, <b>Spain</b>
11:45 - 12:00	Study on the mechanism of plasma-assisted CO <sub>2</sub> methanation over Ru-zeolite catalysts in a DBD <i>operando</i> FTIR cell <b>D. Aceto</b> , F. Azzolina-Jury and C. Henriques	<b>Mr. Domenico Aceto</b> , IST Lisbon, <b>Portugal</b>
12:00 - 12:15	Plasma-liquid catalysis for CO <sub>2</sub> conversion <b>J. Barauna</b> , T. M. García, M. Magureanu and V. I. Parvulescu	<b>Mr. Jairo Barauna</b> , University of Bucharest, <b>Romania</b>
12:15 - 12:30	In-situ water purification method using plasma activated microbubbles for remote environments with limited resources <b>M. Kim</b>	<b>Dr. Minkwan Kim</b> , University of Southampton, <b>UK</b>
12:30 - 12:45	Kinetic mechanisms in CO <sub>2</sub> -N <sub>2</sub> plasmas <b>C. Fromentin</b> , T. Silva, T. C. Dias, E. Baratte, O. Guaitella, O. Biondo and V. Guerra	<b>Mrs. Chloé Fromentin</b> , Technical University of Lisbon, <b>Portugal</b>

29 April 2022

**SICT 2022 / Tribology 2022 Session III: Lubricants and hydrodynamic lubrication**

**Onsite Conference Room Glorias C**

**Session's Chairs:**

**Prof. Giuseppe Carbone, Polytechnic University of Bari, Italy**  
**Prof. Helena Ronkainen, VTT Technical Research Centre, Finland**

08:30 - 09:00	Green Tribology: an appraisal of current status and future needs <b>M. Kalin</b>	<b>Prof. Mitjan Kalin</b> , University of Ljubljana, <b>Slovenia</b>
09:00 - 09:30	The Active Role of Lubricating Grease as a Tribological System- An Energetic Approach <b>E.Kuhn</b>	<b>Prof. Erik Kuhn</b> , Hamburg University of Appl. Sc, <b>Germany</b>
09:30 - 09:45	Development of tribological testing procedure for lubricating oil used in weaving machines. <b>E. Colombo</b> and M. Toscanini	<b>Ms. Eleonora Colombo</b> , R&D downstream research center-Eni SpA, <b>Italy</b> .
09:45 - 10:00	Tribological Performance of Lubricating Greases Composed by Different Bio-based Polymer Thickeners under EHL Conditions <b>S. Vafaei</b> , G. Jacobs, F. König and R. Weberskirch	<b>Mr. Seyedmohammad Vafaei</b> , RWTH Aachen University, <b>Germany</b>
10:00 - 10:30 Coffee Break		
<b>Session's Chairs:</b> <b>Dr. Chen Xiao, Advanced Research Center for Nanolithography, The Netherlands</b>		
10:30 – 10:45	Development of sustainable and effective water-based metalworking fluid for titanium machining <b>E. Benedicto</b> , E.M. Rubio, L. Aubouy and M.M. Marín	<b>Mrs. Elisabet Benedicto</b> , Leitat Technological Center, <b>Spain</b>
10:45 – 11:00	Development of HPDC system for lubricants evaluation <b>L. Batlle</b> , E. Benedicto, D. Cecilia, L. Muntada and W. Ajana	<b>Ms. Laura Batlle</b> , Leitat Technological Center, <b>Spain</b> .
11:00 - 11:15	Study of friction reducers obtained from Sargassum algae: Effect of the structure on the tribological performances of the carbon phases <b>A.Molza</b> , P.Bilas, T.Cesaire, P.Thomas and Y.Bercion	<b>Dr. Audrey Molza</b> , University of the French West Indies and Guiana- Guadeloupe, <b>France</b>
11:15 - 11:30	Mechanism analysis of tribological performance enhancement using MoS <sub>2</sub> and h-BN nanomaterials as nano-additives into poly alpha olefin <b>H. Jiang</b> , X. Hou and K. D. Dearn	<b>Mr. Hua Jiang</b> , Wuhan University of Technology, <b>China</b>
11:30 - 11:45	Tribological response of self-mated Zircaloy-4 under dry and water submerged conditions <b>B. Kumar</b> , D. Kumar and V. Chaudhry	<b>Mr. Bharat Kumar</b> , Indian Institute of Technology Delhi, <b>India</b>
11:45 - 12:00	Performance of non-toxic, corrosion resistance, and lubricious metalworking fluid under machining <b>A.P.S. Lodhi</b> and D. Kumar	<b>Mr. Ajay Lodhi</b> , Indian Institute of Technology Delhi, <b>India</b>

## Virtual Presentations Sessions

The virtual presentations are to be held through the Whova Virtual event solution and can be joined online. Any onsite participant wishing to take part of these virtual sessions, will have a room available for seating.

27 April 2022		
SICT 2022 / Plasma Tech 2022 joint virtual session		
Virtual Conference Session		
Session's Chairs: Prof. Eloisa Sardella, CNR- Institute of Nanotechnology, Italy		
14:00 - 14:30	Biobased functional coatings for cellulosic substrates <b>M.B.Coltelli</b> , L.Panariello, V.Gigante, L. Aliotta, S. Giangrandi, Ahdi Harich, I. Canesi, A. Lazzeri and P. Cinelli	<b>Dr. Maria Beatrice Coltelli</b> , University of Pisa, Italy
14:30 - 14:45	Growth of carbon-based fibrous nanostructures via Chemical Vapor Deposition on half-Heusler alloys A. Manasi, I.G. Aviziotis, A.F.A. Trompeta, A. Ntziouni, S.Deligiannis, A. Alexandratou, E.P. Koumoulos and <b>C.A. Charitidis</b>	<b>Prof. Costas Charitidis</b> , National Technical University of Athens, Greece
14:45 - 15:00	Fabrication of worm-like carbon fibers from Oligomer-rich soot generated by Solution Plasma <b>A. E. Romero Valenzuela</b> , C: Chokradjaroen and N. Saito	<b>Mr. Andres E. R. Valenzuela</b> , Nagoya University, Japan
15:00 - 15:15	Fabrication of Anti-Wetting Coatings for Cold Environments <b>A. Corozzi</b> , F. Veronesi, J. Mora, M. Caruso and M. Raimondo	<b>Dr Alessandro Corozzi</b> , Institute of Science and Technology for Ceramics, National Research Council (ISTEC- CNR), Italy
15:15 - 15:30	Combination of Coagulation, Adsorption, and Ultrafiltration Processes for Organic Matter Removal from Peat Water <b>M Elma</b> , A E Pratiwi, A Rahma, E L A Rampun, Mahmud, C Abdi, R Rosadi, D H Y Yanto and M R Bilad	<b>Prof. Muthia Elma</b> , Lampung mangkurat University, Indonesia
15:30 - 15:45	Control of metal ion release from magnetron sputtered Ag-Cu coatings on textiles <b>S. Sonay Özbay</b> , G.T. Pourian Azar, J. Sharp, G.D. Rajmohan and A. Cobley	<b>Mr. Serdar Sonay Özbay</b> , Coventry University, UK
15:45 - 16:00	Atmospheric pressure plasma superhydrophobic bilayer coatings to limit dairy fouling adhesion <b>M. Saget</b> , N. Nuns, L. Azevedo-Scudeller, G. Delaplace, V. Thomy, Y. Coffinier and M. Jimenez	<b>Mrs. Manon Saget</b> , Univ Lille, France
16:00 - 16:15	Evaluation of galvanic anodes capacity as per NACE TM0190-98 test methods <b>N. Rasheedi</b> and S. Al Mutairi	<b>Mr. Nayif Rasheedi</b> , Saudi Aramco, Saudi Arabia
16:15 - 16:30 Coffee Break		
Session's Chairs: Dr. Maria Beatrice Coltelli, University of Pisa, Italy		
16:30 - 17:00	Breakdown criteria in air: critical analysis and applications <b>O. Eichwald</b> , O. Ducasse and M. Yousfi	<b>Prof. Olivier Eichwald</b> , Paul Sabatier Univ. Toulouse, France
17:00 - 17:15	Active Janus Particles in a Complex Plasma <b>V. Nosenko</b>	<b>Dr. Volodymyr Nosenko</b> , German Aerospace Center DLR, Germany
17:15 - 17:30	Plasma based CO2 Utilization – A Comparison S. Renninger, <b>P. Rößner</b> , J. Stein, M. Lambarth and P. Birke	<b>Mr. Paul Rößner</b> , University of Stuttgart, Germany
17:30 - 17:45	Investigation of Temporal Evolution of Vacuum Arc with Heated Cathode Made of Cerium Dioxide <b>A.D Melnikov</b> , R.A. Usmanov, V.P. Polishchuk, N.N. Antonov, A.V. Gavrikov and G.D. Liziakin	<b>Mr. Anton Melnikov</b> , Joint Institute for High Temperatures of the Russian Academy of Sciences, Russia
17:45 - 18:00	Pb+Ag mixture space separation in crossed fields of background discharge at the LaPlaS setup	<b>Dr. Gennadii Liziakin</b> , Joint Institute for High

	<b>G. Liziakin</b> , N. Antonov, A. Gavrikov, A. Oiler, A. Melnikov, V. Smirnov, R. Timirkhanov, R. Usmanov, L. Volkov and N. Vorona	Temperatures of the Russian Academy of Sciences, <b>Russia</b>
<b>18:00 - 18:15</b>	Optimization of PECVD SiO <sub>x</sub> passivation of GaN for interface charge control <b>O. Richard</b> , V. Aimez and A. Jaouad	<b>Mr Olivier Richard</b> , Sherbrooke University, <b>Canada</b>
<b>18:15 - 18:30</b>	Dynamic properties of dust particles in a DC glow discharge in an external magnetic field <b>A.R. Abdirakhmanov</b> , N.Kh. Bastykova, S.Kh. Kodanova and T.S. Ramazanov	<b>Mr. Assan Abdirakhmanov</b> , Al-Farabi Kazakh National University, <b>Kazakhstan</b>
<b>18:30 - 18:45</b>	Plasma assisted CO <sub>2</sub> dissociation in pure and gas mixture streams at ambient conditions P. Navascués, M Oliva-Ramírez, J. Cotrino, A. R. González-Elipé and <b>A. Gómez-Ramírez</b>	<b>Dr. Ana Gómez-Ramírez</b> , University of Seville, <b>Spain</b>
<b>18:45 - 19:00</b>	Plasma Diagnostics in Glow Discharge Emission Spectroscopy: a Glimpse into the Excitation Processes Involved <b>Z. Weiss</b>	<b>Dr. Zdenek Weiss</b> , Institute of Physics of the Czech Academy of Sciences, <b>Czech Republic</b>

28 April 2022		
Tribology 2022 Virtual Session		
Virtual Conference Session		
Session's Chairs: Prof. Maria Clelia Righi, University of Bologna, Italy		
08:00 – 08:30	Graphene Coatings: A Disruptive Approach to Remarkable Corrosion Resistance R. Singh	Prof. Raman Singh, Monash University, Australia
08:30 - 09:00	How to Slide (almost) without Friction from Boundary to Hydrodynamic Lubrication J. Cayer-Barrio	Prof. Juliette Cayer-Barrio, University of Lyon, France
09:00 - 09:30	Modeling of Lubrication between Parallel Rough Surfaces: Challenges and New Solutions N. Brunetière	Dr. Noel Brunetière, University of Poitiers, France
09:30 - 09:45	Study on the Effect of Water Content on Tribological Performance of Grease S. Y. Chern, W. L. Liu and M. F. Gu	Mr. Wei-Lun Liu, National Formosa University, Taiwan
09:45 - 10:00	Influence of Laser Cladding on the Tribological and Microstructural Properties of Ex-Service Light Rails P. Fasihi, O. Kendall, R. Abrahams, C. Qiu, P. Mutton and W. Yan	Ms. Panahsadat Fasihi, Monash University, Australia
10:15 – 10:30	Study on the Deterioration of Lubricating Oils with Environmental Particles J. H. Horng, J. L. Lin, Y. Y. Lin	Mr. Jin-Long Lin, National Formosa University, Taiwan
10:30 -10:45	Defining a shear hardness: a computational approach R. Capozza and K. J. Hanley	Dr. Rosario Capozza, The University of Edinburgh, UK
10:30 - 11:00 Coffee Break		
Session's Chairs: Prof. Juliette Cayer-Barrio, University of Lyon, France Prof. Jason Stokes, The University of Queensland, Australia		
11:00 - 11:30	Advancing Solid Interfaces and Lubricants by First principles Materials Design M. Clelia Righi	Prof. Maria Clelia Righi, University of Bologna, Italy
11:30 - 12:00	Detection and Prevention of Early Surface Degradation Z. Tian, P. Lu, J. Grundy, S. Wang and R. Wood	Prof. Robert Wood, Univ. of Southampton, UK
12:00 - 12:15	Triboscopy of Carbon Coatings Under Dry Friction Conditions L. Lorenz, F. Härtwig, S. Makowski, M. Krause and A. F. Lasagni	Mr. Lars Lorenz, Dresden Univ. of Technology, Germany
12:15 - 12:30	Hydrostatic load on wavy sealing surfaces D. Huang, X. Yan, R. Larsson and A. Almqvist	Mr. De Huang, Luleå University of Technology, Sweden
12:30 - 12:45	Laser-based processing of polymeric tribological coatings for lightweight applications M. Dahmen, C. Vedder and J. Stollenwerk	Mr. Marius Dahmen, Fraunhofer Institute for Laser Technology (ILT), Germany
12:45 - 13:00	Weighted LASSO feature selection for the analysis of FT-IR spectra applied to relate engine oil degradation patterns P. Pfeiffer, B. Ronai, G. Vorlauffer, N. Dörr and P. Filzmoser	Ms. Pia Pfeiffer, Vienna University of Technology, Austria
13:00 - 13:15	Analysis of the incomplete film in journal bearings using computational fluid dynamic S. Wei , Y. Kligerman, R. Goltsberg and I. Etsion	Dr. Roman Goltsberg, Technion, Israel
13:15 - 13:30	Unexpected low friction property of brass on ta-C in vacuum and atmospheric conditions F. Härtwig, L. Lorenz, S. Makowski, M. Krause, C. Habenicht and A. Fabián Lasagni	Mr. Fabian Härtwig, Fraunhofer Institute for Material and Beam Technology IWS, Germany
13:30 - 14:00 Lunch Break		
Plasma Tech 2022 Virtual Session		
Virtual Conference Session		

<b>Session's Chairs:</b> <b>Prof. Eloisa Sardella, CNR- Institute of Nanotechnology, Italy</b> <b>Prof. Jason H.C. Yang, Feng Chia University, Taiwan</b>		
<b>14:00 - 14:30</b>	Electron Dynamics and Abnormal Target Erosion in RF Magnetron Discharges <b>Qi Hua Fan</b> and Bocong Zheng	<b>Dr. Qi Hua Fan</b> , Michigan State University, <b>USA</b>
<b>14:30 - 15:00</b>	Aerosol assisted plasma deposition of composite coatings: from biomaterials to catalysis <b>F. Palumbo</b> , C. Lo Porto and P. Favia	<b>Dr. Fabio Palumbo</b> , CNR Institute of Nanotechnology, <b>Italy</b>
<b>15:00 - 15:15</b>	Correlation of Excited Chemical Species by Solution Plasma in Cyclic Organic Compounds and the synthesized Carbon-based structure <b>J. Niu</b> , C.Chokradjaroen and N. Saito	<b>Dr. Jiangqi Niu</b> , Nagoya university, <b>Japan</b>
<b>15:15 - 15:30</b>	Reduction Synthesis of High-Entropy Alloy Nanoparticles in the Plasma Ionic Liquid System and their Application to Hydrogen Evolution Reaction <b>G. Lee</b> , V.-T. Nguyen, L. Larina, N.-A. Nguyen, H.-S. Choi and M. Keidar	<b>Prof. Ho Suk Choi</b> , Chungnam National University, <b>Rep. of Korea</b>
<b>15:30 - 15:45</b>	Synthesis of SiC Nanopowder by DC Plasma for Nuclear Fuel Applications <b>G. Cota-Sanchez</b> , D. McDonald, J. Mouris and N. Lee	<b>Dr. German Cota-Sanchez</b> , Canadian Nuclear Laboratories, <b>Canada</b>
<b>15:45 - 16:00</b>	Effect of crosslinker on the wettability and mechanical properties of hydrophobic coatings deposited via atmospheric pressure plasma <b>C. Rendon-Piedrahita</b> , K. Baba, R. Quintana, J. Bardou, R. Heyberger, J. Borek-Donten and P. Choquet	<b>Dr Camilo Rendon Piedrahita</b> , Luxembourg Institute of Science and Technology, <b>Luxembourg</b>
<b>16:00 - 16:15</b>	Solution Plasma for Enhanced Phosphorus Doping in TiO <sub>2</sub> Nanoparticles <b>C. Chokradjaroen</b> , J. Niu and N. Saito	<b>Dr. Chayanaphat Chokradjaroen</b> , Nagoya University, <b>Japan</b>
<b>16:15 - 16:30 Coffee Break</b>		
<b>Chairs:</b> <b>Prof. Olivier Eichwald, Paul Sabatier Univ. Toulouse, France</b> <b>Dr. Fabio Palumbo, CNR Institute of Nanotechnology, Italy</b> <b>Dr. Qi Hua Fan, Michigan State University, USA</b>		
<b>16:30 - 17:00</b>	Adaptive Plasmas and Recent Developments in Plasma Medicine <b>M. Keidar</b>	<b>Prof. Michael Keidar</b> , George Washington Univ., <b>USA</b>
<b>17:00 - 17:30</b>	The plasma produced long-lived RONS as allies in biomedicine and food storage. <b>E. Sardella</b>	<b>Prof. Eloisa Sardella</b> , CNR-Institute of Nanotechnology, <b>Italy</b>
<b>17:30 - 18:00</b>	With Plasma from Medicine to Hydrogen <b>K.D. Weltmann</b> , T.v. Woedtke, J.F. Kolb, R. Brandenburg, T. Gerling, S. Bekeschus, R. Bansemer, V. Hahn, J. Ehlbeck, H. Brust, K. Zocher, V. Brüser, A. Kruth and R. Clemen	<b>Prof. Klaus-Dieter Weltmann</b> , Leibniz Institute for Plasma Science and Technology e.V. <b>Germany</b>
<b>18:00 - 18:15</b>	Plasma-Induced Graft Polymerization of Polyethylenimine onto Poly-caprolactone Composite Membrane for Heavy Metal Pollutants Treatment in Industrial Wastewater S-L. Tu and <b>J.H.C. Yang</b>	<b>Prof. Jason H.C. Yang</b> , Feng Chia University, <b>Taiwan</b>
<b>18:15 - 18:30</b>	Application of continuous flow plasma brushes for inactivation of antibiotics from communal and industrial wastewaters <b>W. Babinska</b> , A. Motyka-Pomagruk, D. Terefinko, M. Caban, P. Jamroz, P. Pohl, E. Lojkowska, W. Sledz and A. Dzimitrowicz	<b>Mrs. Weronika Babińska</b> , University of Gdansk, <b>Poland</b>
<b>18:30 - 18:45</b>	The Physical Effects of Plasma Medicine on Cells: Radio Frequency Stimulated Intercellular and Intracellular Mechanical Waves <b>L. Lin</b> , X. Yao, D. Yan and M. Keidar	<b>Dr. Li Lin</b> , The George Washington University, <b>USA</b>
<b>18:45 - 19:00</b>	Antibacterial, biodegradable nanohybrids for therapy of chronic wounds. <b>A. Manakhov</b> , E. Permyakova and A. Solovieva	<b>Mr. Anton Manakhov</b> , Research Institute of Clinical and Experimental Lymphology <b>Russia</b>



29 April 2022		
Tribology 2022 Virtual session: Biotribology/ Green Tribology		
Virtual Conference Session		
Session's Chairs: Prof. Robert Wood, University of Southampton, UK Prof. Jason Stokes, The University of Queensland, Australia		
09:00 - 09:30	Learning from Nature: Discovering and Designing Bio-inspired Solutions in Tribology D. Dini	Prof. Daniele Dini, Imperial College London, UK
09:30 - 10:00	Soft tribology, biotribology and viscoelastic lubrication: interpretation and application J.R. Stokes and Y. Xu	Prof. Jason Stokes, The University of Queensland, Australia
10:00 - 10:30 Coffee Break		
Session's Chairs: Prof. Jason Stokes, The University of Queensland, Australia		
10:30 – 11:00	Using tribology to prevent skin injury and improve comfort M.A. Masen, J. Hayes, R.D. Jobanputra, K.K. Yap, M. Murali and Z. Tan	Prof. Marc Marsen, Imperial College London, UK
11:00- 11:30	How to go green in tribology? Sustainability and resource efficiency do matter in the 21st century! C. Gachot	Prof. Carsten Gachot, Vienna University of Technology, Austria

## SICT 2022 / PlasmaTech 2022 / Tribology 2022 Joint Conferences Posters

Posters are being displayed through the Virtual event solution.

Discussions are to be done through the system chat features available to the attendees.

N.	Poster Title	Author, Affiliation, Country
1.	Modeling of surface morphology kinetics and nanostructures formation during binray thin film deposition <b>A. Galdikas</b> and G. Kairaitis	<b>Prof. Arvidas Galdikas</b> , Kaunas University of Technoogy, <b>Lithuania</b>
2.	Development of functional coatings with anti-abrasion and anti-icing properties <b>J. P. Sousa</b> and Y. Kolen'ko	<b>Dr. Juliana Sousa</b> , International Iberian Nanotechnology Laboratory, <b>Portugal</b>
3.	Functional nanomaterial-based coatings for automotive industry <b>C. Ponte</b> and J. P. Sousa	<b>Dr. Clara Ponte</b> , International Iberian Nanotechnology Laboratory, <b>Portugal</b>
4.	Synthesis and characterization of GaN/ReS <sub>2</sub> , ZnS/ReS <sub>2</sub> and ZnO/ReS <sub>2</sub> core/shell nanowire heterostructures E. Butanovs, A. Kuzmin, S. Piskunov, K. Smits, A. Kalinko and <b>B. Polyakov</b>	<b>Dr. Boris Polyakov</b> , University of Latvia, <b>Latvia</b>
5.	Surface chemical and physical modification of PAM film induced by ion-beam irradiation for liquid crystal alignment <b>D. W. Lee</b> , J. H. Won, D. H. Kim, J. Y. Oh and D.-S. Seo	<b>Mr. DongWook Lee</b> , Yonsei University, <b>Rep of Korea</b>
6.	Self-improving of Protective Thin Films Based on Perhydropolysilazane <b>E. Shmagina</b> , M. Danilson, V. Mikli and S. Bereznev	<b>Ms. Elizaveta Shmagina</b> , Tallinn University of Technology, <b>Estonia</b>
7.	Fabrication Multilayer Nanocoating for Marine Applications <b>M. Caruso</b> , A. Corozzi, F. Veronesi, M. Raimondo, V. Piazza, F. Garaventa and F. Castelli	<b>Mrs. Maria Caruso</b> , CNR-ISTEC Institute of Science and Technology for Ceramic, <b>Italy</b>
8.	Steam-corrosion resistance of polycrystalline Ag thin optical coatings improving by grain-boundary segregation effect <b>G. Marchii</b> , D. Samsonov, I. Tereschenko, A. Guba and E. Mukhin	<b>Mr. Georgiy Marchiy</b> , Ioffe Institute, <b>Russia</b>
9.	The Effect of Carbon Nanotubes on the Corrosion Resistance of Waterborne Polyurethane-Acrylate Coatings <b>J. T. Corredor</b> and L.F. Giraldo	<b>Dr. Jeaneth Corredor</b> , Antioquia University, <b>Colombia</b>
10.	Adhesion of polyurethane coatings with powder fillers on composite substrates P. Mayer, <b>H. Kaplon</b> , A. Dmitruk, M. Lubecki, K. Rosochacka and P. Kus	<b>Mrs. Honorata Kaplon</b> , Wrocław University of Science and Technology, <b>Poland</b>
11.	Interactions of poly(ethyl-cyanoacrylate) with surfaces of aluminium oxide and titanium oxide <b>P. Moritz</b> , L. Wegewitz and W. Maus-Friedrichs	<b>Mr. Philipp Moritz</b> , Clausthal University of Technology, <b>Germany</b>
12.	Light Activated Inorganic Agents - the next generation of photocatalytic paints R. Bucureşteanu, V. Chihaia, B. Cojocaru, M. Ioniţă, A. Ficai, <b>L.M. Ditu</b> and G. Mihăescu	<b>Prof. Lia-Mara Ditu</b> , University of Bucharest, <b>Romania</b>
13.	Colloidal and Thermal stability of Hybrid Waterborne Polyurethane Dispersions Prepared by Grafting Method <b>L. F. Giraldo</b> , Jeaneth T. Corredor, Esneyder Ruiz, Victor H. Orozco	<b>Prof. Luis Fernando Giraldo</b> , University of Antioquia, <b>Colombia</b>
14.	Antibacterial SnO <sub>2</sub> coatings applied on Ti6Al7Nb alloy for applications in bone surgery <b>M. Basiaga</b> , W. Walke and J. Lisoń	<b>Prof. Marcin Basiaga</b> , Silesian University of Technology, <b>Poland</b>
15.	Ti13Zr13Nb alloy modified of tin dioxide by means of nanoPVD and ALD methods for an implant used in the skeletal system <b>W Walke</b> , M. Basiaga and K. Goldsztajn	<b>Prof. Witold Walke</b> , Silesian University of Technology, <b>Poland</b>
16.	Optimization of laser parameters for the injection moulds cleaning R. Santos, I. Marcelino, T. Ferreira, L. Pereira, F.M. Costa, <b>N. M. Ferreira</b>	<b>Dr. Nuno Ferreira</b> , Aveiro University, <b>Portugal</b>
17.	Design of polymeric coatings based on pH-sensitive acrylic terpolymers with potential application in the treatment of colonic diseases. <b>L.M Suárez Giraldo</b> , V. Orozco López and L.F Giraldo Morales	<b>Ms. Lina M. Suarez Giraldo</b> , University of Antioquia, <b>Colombia</b>



18.	Enhancing sensitivity and reducing unspecific adsorption in fiber-attached polymer hydrogels <b>A. Luongo</b> , T. Brandstetter and J. Rühle	<b>Dr. Anna Luongo</b> , University of Freiburg, <b>Germany</b>
19.	The Influence of Yarn Fineness in Layer Interchanging Double Cloth on Woven Fabric Roughness <b>I. Schwarz</b> , T. Badrov, S. Kovačević	<b>Prof. Ivana Schwarz</b> , University of Zagreb, <b>Croatia</b>
20.	Woven Fabric Roughness Conditioned by Weave <b>S. Brnada</b> , A. Kalazić, S. Sabljak and T. Kaurin	<b>Dr. Snjezana Brnada</b> , University of Zagreb, <b>Croatia</b>
21.	Plasma vortices in the near Earth environment <b>K. Elbakidze</b>	<b>Dr. Khatuna Elbakidze</b> , Tbilisi State University, <b>Georgia</b>
22.	A plasma diagnostic package for spacecrafts with electric propulsion systems <b>V. Schneider</b> , T. Trottenberg, H. Kersten, J. Laube, H. Henkel and R. Wimmer-Schweingruber	<b>Dr. Viktor Schneider</b> , University Kiel, <b>Germany</b>
23.	Experimental Observation of plasma parameter and photoresist ashing in an inductively coupled plasma sources with MRWPT antenna <b>J-H. Kim</b> , Y-H. Hong and C-W. Chung	<b>Dr. Ju Ho Kim</b> , Hanyang University, <b>Rep. of Korea</b>
24.	Experimental observation of hysteresis in a neon inductively coupled plasma at low pressure Y-H. Hong, T-W. Kim, M-Y. Lee, J-H. Kim and C-W. Chung	<b>Mr. Young-Hun Hong</b> , Hanyang University, <b>Rep. of Korea</b>
25.	Spatial and Temporal Characterization of NO and OH Concentration in a Nanosecond Pulsed Surface Dielectric Barrier Discharge Plasma Using Picosecond Laser Induced Fluorescence <b>L. Ibba</b> , P. F. Ambrico, F. Avino and I. Furno	<b>Mr. Lorenzo Ibba</b> , EPFL University, <b>Switzerland</b>
26.	Dielectric barrier discharge plasma reduction of iron oxides <b>V. Udachin</b> , S. Dahle and W. Maus-Friedrichs	<b>Mr. Viktor Udachin</b> , Clausthal University of Technology, <b>Germany</b>
27.	Deposition in silicon trenches and porous substrate using Bipolar High power impulse magnetron sputtering S. Atmane, N. Rochdi and <b>A. Caillard</b>	<b>Mr. Caillard Amael</b> , University of Orléans, <b>France</b>
28.	Plasma / liquid interactions during low pressure plasma sputtering deposition of Pt nanoparticles on liquid glycerol <b>S. Atmane</b> , P. Brault and A. Caillard	<b>Mrs. Soumya Atmane</b> , University of Orléans, <b>France</b>
29.	Kinetic Monte-Carlo modeling of SiN:H thin film deposition by PECVD on complex substrates: characterization of air-connected porosity and improvement by ion beam assisted deposition <b>J. Müller</b> , P. Moskovkin and S. Lucas	<b>Dr. Jerome Muller</b> , University of Namur, <b>Belgium</b>
30.	The influence of the RF- power on the photocatalytic properties of thin PE-ALD ZnO films deposited at room temperature <b>D. Jardas</b> , R. Peter, I. Turel, M. Petravić and A. Omerzu	<b>Ms. Daria Jardas</b> , University of Rijeka, <b>Croatia</b>
31.	Influence of adatoms on the titanium nano-cone formation during helium ion bombardment <b>F. Sanchez</b> , L. Marot, R. Antunes, R. Steiner, J. Spicher, P. Lattner, M. Kisiel, D. Mathys, E. Meyer, M. Astasov-Frauehoffer, I. Hauser-Gerspach, S. Kühl, J. Köser, R. Wagner, J. Hofstetter and K. Mukaddam	<b>Mr. Fabien Sanchez</b> , University of Basel, <b>Switzerland</b>
32.	Properties of (W,Zr)B <sub>2</sub> -z Protective Coatings Deposited by RF Magnetron Sputtering Method <b>T. Mościcki</b> , R. Psiuk and J. Chrzanowska-Giżyńska	<b>Dr. Tomasz Moscicki</b> , Institute of Fundamental Technological Research PAS, <b>Poland</b>
33.	Adsorption of Organic Components from Fluid Mixtures on Cold Plasma Coated Aerogels in Supercritical Fluid Chromatography: Experiment and Simulation <b>I. Jung</b> , B. Schroeter, P. Gurikov and I. Smirnova	<b>Mrs. Isabella Jung</b> , Hamburg University of Technology, <b>Germany</b>
34.	Photoreactive pressure sensitive adhesives - bacterial and fungal properties <b>K. Mozelewska</b> and P. Niezgoda	<b>Ms. Karolina Mozelewska</b> , West Pomeranian University of Technology in Szczecin, <b>Poland</b>
35.	Decontamination of Drosophila suzukii Infested Blueberries with Low-pressure Cold Plasma Treatment <b>P. Starič</b> , N. Cvelbar Weber, M. Resnik, K. Vogel Mikuš and I. Junkar	<b>Ms. Pia Staric</b> , Jožef Stefan Institute, <b>Slovenia</b>

36.	Deposition of microencapsulated essential oil extracted from Neem seeds on the surface of plasma pretreated cellulose knitwear <b>T. Perinović</b> , A. Ludaš and S. Ercegović Ražić	<b>Ms. Tihana Perinović</b> , University of Zagreb Faculty of Textile Technology, <b>Croatia</b>
37.	Implementation of a Non-Thermal Atmospheric Pressure Plasma for Eradication of Plant Pathogens from a Surface of Economically Important Seeds <b>J. Orlowski</b> , A. Motyka-Pomagruk, A. Dzimitrowicz, W. Babinska, D. Terefinko, M. Rychlowski, M. Prusinski, P. Pohl, E. Lojkowska, P. Jamroz and W. Sledz	<b>Mr. Jakub Orlowski</b> , University of Gdansk, <b>Poland</b>
38.	Slip Resistance Improvement for Shoe Outsoles through Atmospheric-Pressure Plasma Treatments R. Múgica-Vidal, I. Muro-Fraguas, A. Sainz-García, E. Sainz-García, A. González-Marcos and <b>F. Alba-Elías</b>	<b>Dr. Fernando Alba-Elías</b> , University of La Rioja, <b>Spain</b>
39.	Tribological Performance of 2D Nano-coated Steel Surfaces <b>M.J.G. Guimarey</b> , M. Hadfield and A. Abdelkader	<b>Dr. María J. García Guimarey</b> , Bournemouth University, <b>UK</b>
40.	Effect of electrochemistry on tribochemical wear of monocrystalline silicon <b>C. Xiao</b> , F-C. Hsia, B. Weber and S. E. Franklin	<b>Dr. Chen Xiao</b> , Advanced Research Center for Nanolithography, <b>The Netherlands</b>
41.	Tribological and corrosion properties of coating systems dried by infrared radiation I. Stojanović, <b>L. Turkalj</b> , I. Cindrić, I. Juraga and D. Rakela Ristevski	<b>Mr. Lovro Turkalj</b> , University of Zagreb, <b>Croatia</b>
42.	Nonlinear Dynamic Analysis of Slot-Type Externally Pressurized Air Bearing System <b>C.C. Wang</b>	<b>Prof. Cheng-Chi Wang</b> , National Chin-Yi University of Technology, <b>Taiwan</b>
43.	Characterization and simulation of tribological behaviour for WC spherical surfaces with AlTiSiN coating <b>M. Marqués</b> , A.Claver, J.A.García, D. Salcedo, A. Tellez and I. Quintana	<b>Mr. Mikel Marqués</b> , Public University of Navarre, <b>Spain</b>
44.	Optimized surface engineering solutions for glass mould industry <b>J. Costa</b> , J.P. Dias and A. Cavaleiro	<b>Dr. João Costa</b> , Pedro Nunes Institute, <b>Portugal</b>
45.	Tool condition monitoring of self-lubricating nitride coatings to improve the lifetime of cutting tools in the manufacturing industry <b>J. Perdigoto</b> , B. Martins, J. Dias, D. Silva, T. Todo Bom, R. Curado, N. Alves, P. Rodrigues and D. Fonseca	<b>Dr. José Perdigoto</b> , Pedro Nunes Institute, <b>Portugal</b>
46.	Challenges on Protein Bioencapsulation in Transparent Nanoporous carbothermal reduction of mill scales formed on steel billets during continuous casting <b>S.M. Espinoza Suarez</b> , L.E. Borja-Castro, M. I. Valerio-Cuadros, A. Bustamante Domínguez, H.A. Cabrera-Tinoco, E. Huaman, R.A. Valencia-Bedregal, X. Zhao and L. De Los Santos Val-ladares	<b>Ms. Silvia M.E. Suarez</b> , National Mayor de San Marcos University, <b>Peru</b>
47.	Study of Mill Scale Surface Morphology and Iron Oxide Layers Obtained During Quenching of Steel Billets in The Continuous Casting <b>R. A. Valencia-Bedregal</b> , L. E. Borja-Castro, M. I. Valerio-Cuadros, S. M. Espinoza Suarez, A. G. Villasante Miranda, E. Y. Huaman Enriquez, A. Bustamante Dominguez and L. De Los Santos Valladares	<b>Mr. Renato A.V. Bedregal</b> , National Mayor de San Marcos University, <b>Peru</b>
48.	Surface and structural characterization of recycled HRB335 steel from black slags obtained from Electric Arc Furnace <b>E.Y. Huaman Enriquez</b> , S.M Espinoza Suarez, A.G. Villasante Miranda L.E. Borja-Castro, R.A. Valencia-Bedregal, M.I. Valerio-Cuadros, A. Bustamante Domínguez, X. Zhao, D. Zhang and L. De Los Santos Valladares	<b>Ms. Evelin Yenifer Huaman Enriquez</b> , National Mayor de San Marcos University, <b>Peru</b>
49.	Surface morphology of an Extruded Q235 Steel Rod Fabricated from Rolling Billets Scales <b>A. G. Villasante Miranda</b> , E. Y. Huaman Enriquez, L.E. Borja Castro, R. Valencia Bedregal, S.M. Espinoza Suarez, M.I. Valerio Cuadros, X. Zhao, A. Bustamante Dominguez and L. De Los Santos Valladares	<b>Mr. Alejandro Germán Villasante Miranda</b> , National Mayor de San Marcos University, <b>Peru</b>
50.	Morphological and Mineral characterization of Mill Scales obtained by rolling process <b>L.E. Borja Castro</b> , J. Kargin, R.A. Valencia Bedregal, S.M Espinoza Suarez, A.G. Villasante Miranda, M.I. Valerio-Cuadros, A. Bustamante Domínguez and L. De Los Santos Valladares	<b>Mr. Luis Borja Castro</b> , National Mayor de San Marcos University, <b>Peru</b>

<b>51</b>	Hydrophobic concrete based on hydrophobic sand <b>A. Seralin</b> , A. Kurbanova, T. Khassenova, O. Ualibek, G. Sugurbekova and O. Toktarbaiuly	<b>Mr. Aidar Seralin</b> , Nazarbayev University, <b>Kazakhstan</b>
<b>52</b>	A microfluidic non-thermal plasma reactor equipped with inline emission spectroscopy. <b>P. Roszkowska</b> , A. Slater, J. Walsh, A. Dickenson and T. Easun	<b>Ms. Patrycja Roszkowska</b> , University of Liverpool, <b>UK</b>
<b>53</b>	Remotion of the degraded coating of the polymer injection moulds for their reuse. <b>R. Santos</b> , T. Ferreira, L. Pereira, F. Costa and N. M. Ferreira	<b>Mr. Rodrigo Santos</b> , University of Aveiro, <b>Portugal</b>

# Onsite Presentations

# **SICT 2022 / Plasma Tech 2022 / Tribology 2022 Joint Plenary Onsite session**

# Continuous to Nanoflake MoS<sub>2</sub> Single-Layers: Characterization and Sensing Applications using Optical Spectroscopies

A. de Andrés, S. Cortijo-Campos, L. Álvarez-Fraga, C. Prieto

Instituto de Ciencia de Materiales de Madrid, CSIC, Cantoblanco 28049 Madrid, Spain.

## Abstract:

Raman micro-spectroscopy is probably the most commonly used technique to characterize most 2D materials. However, defects, strain and doping produce modifications in the phonons that can blur a straightforward interpretation of the Raman spectra. MoS<sub>2</sub> is one of the most studied bidimensional transition metal dichalcogenides because of its excellent light absorption and emission properties. 2D-MoS<sub>2</sub> has excellent performance in a wide variety of applications related to its optical characteristics, from photodetectors [1] to biosensors [2]. Photoluminescence (PL) is a key feature for most applications but its quantum yield is maximum only for single-layers, due to the direct to indirect bandgap transition occurring from one to two layers, being thus undetectable for few-layer samples. Therefore, high quality single-layers are required in many cases. Here we will present how the combination of Raman, PL and optical transmittance micro-spectroscopies can provide detailed information on the characteristics of 2D-MoS<sub>2</sub> over a wide range of sizes (from continuous films to nanoflakes) obtained by different techniques (CVD, PVT and mechanical exfoliation). The complex Raman spectrum of 2D-MoS<sub>2</sub> is analyzed in detail re-

vealing double resonance processes and the impact of the substrate and the growth technique [3].

Micro-transmittance spectroscopy is an optimum tool to reveal the changes in the electronic structure as the number of layers is reduced and much less sensitive to defects/doping than PL. Combining these optical techniques we show that it is possible to reveal relevant characteristics of single and few-layers MoS<sub>2</sub> nanoflakes obtained by CVD and PVT and their dependence on size [4]. The enhancement of the PL signal for single and few-layer MoS<sub>2</sub> is a crucial aspect for sensing applications. We show that Ga and MoO<sub>3</sub> nanoparticles on 2D-MoS<sub>2</sub> flakes provide PL and Raman enhancement by SERS and charge transfer processes [5]. Finally, we show that the modifications of the PL band of MoS<sub>2</sub> single-layer PVT flakes modified with a thiolated DNA probe complementary to the target biomarker can be used as a specific probe to detect the biomarker miRNA21c related to breast cancer [6].

**Keywords:** 2D-materials, TMDC, SERS, Raman phonons, nanoflakes, optical sensing, biomedical applications.

## References:

1. JF. Gonzalez Marin, et al. MoS<sub>2</sub> photodetectors integrated with photonic circuits. *npj 2D Mater Appl* 3, 14 (2019).
2. K. Kalantar-zadehn, J. Zhen Ou, Biosensors Based on Two-Dimensional MoS<sub>2</sub>, *ACS Sens.* 1, 5, (2016)
3. S. Cortijo-Campos, P. Kung, C. Prieto and A. de Andrés. "Forbidden and second order phonons in Raman spectra of single and few-layer MoS<sub>2</sub> close to C exciton resonance ». *J. Phys Chem C* 125, 23904, (2021)
4. S. Cortijo-Campos, C. Prieto and A. de Andrés. "Size effects in single- and few-layer MoS<sub>2</sub> nanoflakes". Submitted to *Nano-materials* (2022).
5. S. Catalán Gómez, S. Garg, A. Redondo Cubero, N. Gordillo, A. de Andrés, F. Nucciarelli, S. Kim, P. Kung, J.L. Pau, "Photoluminescence enhancement of monolayer MoS<sub>2</sub> using plasmonic gallium nanoparticles" *Nanoscale Advances* 1, 884 (2019)
6. S. Catalán-Gómez, et al. "Breast cancer biomarker detection through the photoluminescence of epitaxial monolayer MoS<sub>2</sub> flakes" *Scientific Rep.* 10, 16039 (2020)

# Post-Thermal Spray Coating Surface Modification and Potential Industrial Applications

A. Amanov<sup>1,2,\*</sup>, Y.S. Pyun<sup>2</sup>

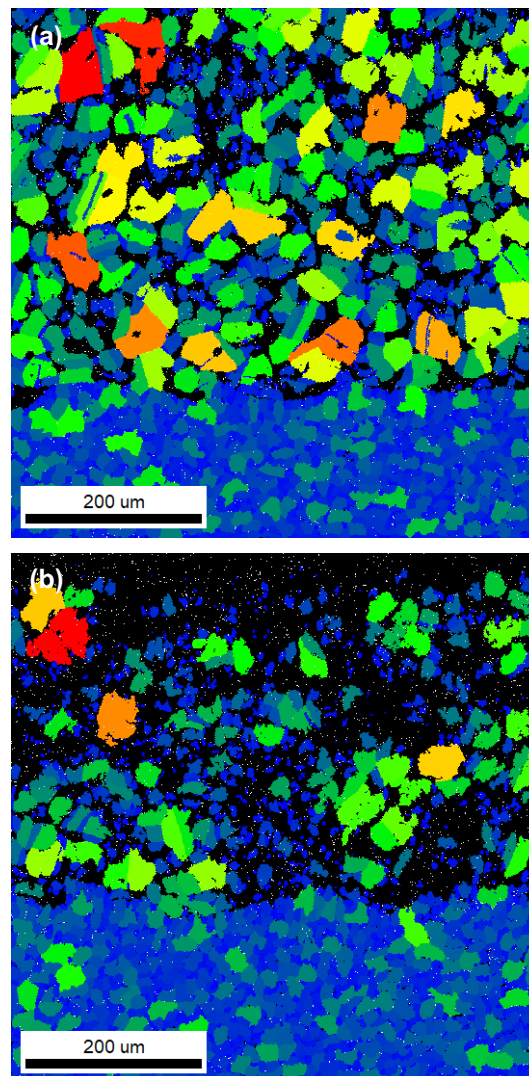
<sup>1</sup> Department of Mechanical Engineering, Sun Moon University, Asan, Korea

<sup>2</sup> Department of Fusion Science and Technology, Sun Moon University, Asan, Korea

## Abstract:

Silicon carbide (SiC) coating is an advanced non-oxide ceramic having unique mechanical, chemical, thermal and electronic properties [1]. It is commonly used in various high-temperature applications such as nuclear, aerospace, optical, etc., where its hardness and fracture toughness play the most important and critical role [2]. In fact, it is possible to achieve a good SiC coating by controlling the main parameters of thermal spray parameters that have an essential effect on its quality and overall properties. Over the last decades, a lot of researchers used several surface modification technologies to improve the mechanical and tribological properties and also fatigue behavior of advanced ceramic coatings. For example, LSP (laser shock peening), PLM (pulsed laser machining), IB (ion bombardment), MSP (micro-shot peening) and many other surface treatment technologies were applied to advanced ceramic coatings and demonstrated promise findings. In this study, an ultrasonic nanocrystal surface modification (UNSM) is considered one of the surface severe plastic deformation ( $S^2PD$ ) methods that has a plenty of advantages over other surface modification technologies. This technology was applied to a SiC coating sprayed by high velocity oxygen fuel (HVOF) method. Figure 1 shows the cross-sectional electron backscattered diffraction (EBSD) images of the as-sprayed and UNSM-treated SiC coatings. It can be observed that UNSM was able to form a compacted and nanostructured surface layer, whose surface hardness significantly increased. Current study provides more comprehensive discussion on the improvement of tribological and fatigue properties of SiC coating. Hence, it is expected that the UNSM can be considered as an alternative promising post-TS coating technology for potential high-performance applications.

**Keywords:** SiC, HVOF, microstructure, grain size, tribology, fatigue, industrial applications.



**Figure 1:** Cross-sectional electron backscattered diffraction (EBSD) images of the as-sprayed (a) and UNSM-treated (b) SiC coatings

## References:

1. Davis, J.R. (2004) Handbook of Thermal Spray Technology, ASM International, Materials Park, OH, USA.
2. Xie, Y., Hawthorne, H. (1999) The damage mechanisms of several plasma-sprayed ceramic coatings in controlled scratching, *Wear*, 233–235, 293–305.



# The Energy Balance of Nano- and Microparticles during Plasma Processing

H. Kersten<sup>1\*</sup>

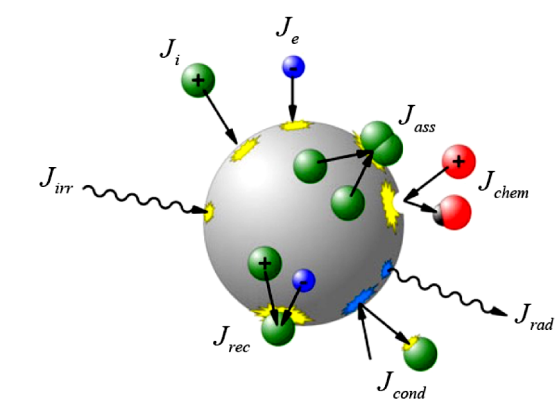
<sup>1</sup> Institute of Experimental and Applied Physics, Christian-Albrechts-University, Kiel, Germany

## Abstract:

The origin of particle production during plasma processing in respect to the formation of nano-composite materials are (i) the generation of large molecules, mesoscopic clusters and nanoparticles in the plasma bulk by chemically reactive gases, and (ii) the formation and incorporation of nanoparticles at surfaces (target, substrate) by means of plasma-wall interaction (e.g. sputtering, thin film deposition). The plasma promotes particle formation by excitation and ionization as well as by dissociation and reaction of the involved species in the gas phase, in particular, by electron collisions. Typical examples are plasma polymerization and thin film deposition in precursor-containing plasma enhanced chemical vapor deposition (PECVD) processes [1] or sputtering of metal clusters [2].

Determination and understanding of energy fluxes to nano- or microparticles, which are confined in process plasmas, is highly desirable because the energy balance results in an equilibrium particle temperature which affects the nanoparticle properties [3]. A simple balance model is used to estimate the energy fluxes between plasma and particles on the basis of measured plasma parameters, see Figure 1. Addition of molecular gases to rare gas plasma results in additional heating of the particles due to molecule recombination. The several contributions  $J$  which affect the particle temperature are discussed in detail. Of special interest are the energy transfer by metastables and the recombination of charge carriers and atoms, respectively, at the particle surface which can result in remarkable heating and subsequent crystallization of nanoparticles related to the deposition of nano- crystalline thin films [4]. By changing the gas composition, the microstructure of the particles can be controlled from mostly amorphous to diamond cubic crystalline.

**Keywords:** dusty plasma, nanoparticle growth, energy balance, plasma diagnostic, plasma-particle interaction



**Figure 1:** Energy fluxes towards and from a particle in plasma determine its temperature.

## References:

1. Hollenstein, C., Schwarzenbach, W., Howling, A.A., Courteille, C., (1996) Anionic clusters in dusty hydrocarbon and silane plasmas, *J. Vac. Sci. Technol.* 14, 535-539.
2. Kratochvil, J., Kuzminova, A., Kylian, O., Biederman, H., (2015) Comparison of magnetron sputtering and gas aggregation nanoparticle source used for fabrication of silver nanoparticle films, *Surf. Coat. Technol.* 275, 296-302.
3. Maurer, H., Kersten, H., (2011) On the heating of nano- and microparticles in process plasmas, *J. Phys. D: Appl. Phys.* 44, 174029.
4. Kramer, H.J., Anthony, R.J., Mamunuru, M., Aydil, E.S., Kortshagen, U.R., (2014) Plasma-induced crystallization of silicon nanoparticles, *J. Phys. D: Appl. Phys.* 47, 075202.



# Graphene nanowalls grown on stainless steel by inductively coupled plasma chemical vapor deposition

Enric Bertran-Serra<sup>1,2</sup>, Stefanos Chaitoglou<sup>1,2</sup>, Roger Amade Rovira<sup>1,2</sup>,  
Arevik Mushegyan-Avetishyan<sup>1,4</sup>, Islam Alshaikh<sup>1,2</sup>, Luis Fernando Pantoja-Suarez<sup>1,3</sup>,  
José Luis Andújar Bella<sup>1,2</sup>, Angel Perez-del-Pino<sup>4</sup>, Eniko Georgy<sup>5</sup>

<sup>1</sup> ENPHOCAMAT, Dep. Applied Physics, Univ. Barcelona, Martí i Franquès 1, E-08028 Barcelona, Spain

<sup>2</sup> Institute of Nanoscience and Nanotechnology (IN2UB), Universitat de Barcelona, E-08028 Barcelona, Spain

<sup>3</sup> Dep. of Materials, Mechanical Engineering Faculty, Escuela Politécnica Nacional, 170525 Quito, Ecuador

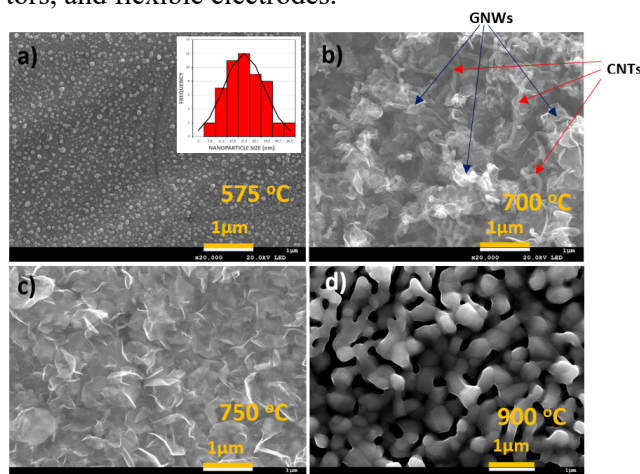
<sup>4</sup> ICMAB-CSIC), Campus UAB, E-08193 Bellaterra, Spain

## Abstract:

It has become clear that it is possible to grow flat graphene nanostructures from the gas phase on planar substrates, but one of the keys to success to produce a very large specific surface in a reduced space is the use of 3D carbon nanostructures (specifically, vertical graphene nanowalls, VGNWs) over a planar substrate, as a growth template for the deposition of electrochemically active materials, as for example, transition metal oxides (TMO). Vertical graphene nanowalls, also known as graphite petal-like, vertical graphene flakes, or vertical graphene, can achieve a very large specific surface area of 1100 m<sup>2</sup>/g, which is comparable to or greater than that of carbon nanotubes, which is reference material for its use in high-performance supercapacitors or in other energy-related applications requiring a large active surface area. Vertical graphene nanowalls also exhibit high vertical and in-plane electrical conductivity when grown on metal electrodes, which benefits their use in electrochemical applications. We are going to focus the discussion on the growth of VGNWs on flexible stainless steel substrates (SS310), in principle suitable for applications to electrodes of electrochemical systems (batteries, supercapacitors, catalysts), and specifically we will discuss the effect of growth temperature on morphological and structural characteristics of graphene nanowalls grown on these flexible substrates. For that, we present the results of the analysis of the Raman spectra and field emission scanning electron microscopy (FE-SEM) of the VGNWs/SS310 samples obtained by inductively coupled plasma chemical vapor deposition (ICP-CVD), from methane as a carbon precursor, in a wide range of temperatures (575 to 900°C) (Fig. 1). Although the nanostructures of graphene nanowalls reported to date, in general, are based on multilayered graphene, what we want to highlight is the effect of temperature on the number of atomic layers of GNW, and that in the environment of 700-750°C, in the

plasma conditions that we have explored, vertical graphene nanowalls are bilayer, which directly affects the magnitude of the VGNW specific surface.

**Keywords** Graphene, nanowalls, nanoflakes, 3D vertical graphene nanowalls, graphite petal-like, VGNW, bilayer and few-layer graphene, carbon nanostructures, SS310, inductively coupled plasma chemical vapor deposition, methane, Raman, nanostructure morphology, supercapacitors, and flexible electrodes.



**Figure 1:** FE-SEM images of carbon nanostructures corresponding to different growth temperatures on stainless steel.

## References:

1. S Chaitoglou and E Bertran, *Effect of temperature on graphene grown by chemical vapor deposition*, J Mater Sci 52(2017)8348
2. R Amade, A Muyshegyan-Avetisyan, J Martí-González, A Pérez del Pino, E György, E Pascual, JL Andújar and E Bertran-Serra, *Super-Capacitive Performance of Manganese Dioxide/Graphene Nano-Walls Electrodes Deposited on Stainless Steel Current Collectors*, Materials, 12, 483 (2019)

3. Arevik Musheghyan, *Synthesis and characterization of multilayer graphene nanostructures*, Doct. Thesis, Barcelona University, (2019); <http://diposit.ub.edu/dspace/handle/2445/142243>

**SICT 2022 Session I:  
Surfaces and Coatings processing /  
Characterization / Properties  
Multifunctional composite and  
hybrid coatings**

# A comparative study of Ir(dmpq)2(acac) doped CBP, mCP, TAPC and TCTA for phosphorescence OLEDs

D. Tselekidou<sup>1</sup>, L. Panagiotidis<sup>1</sup>, K. Papadopoulos<sup>1</sup>, V. Kyriazopoulos<sup>2</sup>, M. Gioti<sup>1,\*</sup>

<sup>1</sup> Nanotechnology Lab LTFN, Department of Physics, Aristotle University of Thessaloniki, GR-54124, Thessaloniki, Greece

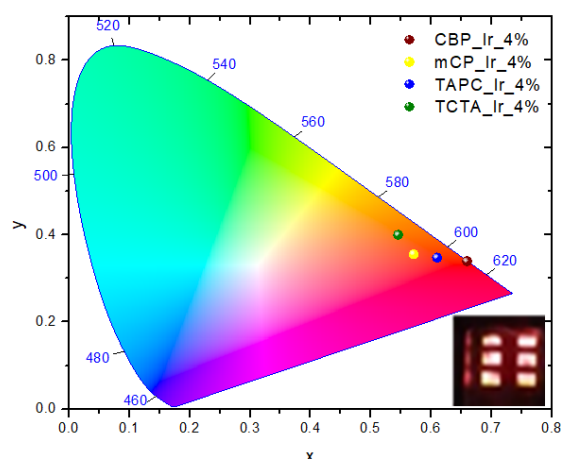
<sup>2</sup> Organic Electronic Technologies P.C. (OET), Antoni Tritsi 21B, GR-57001 Thessaloniki, Greece

## Abstract:

Organic light-emitting diodes (OLEDs) are generally considered as an emerging technologies for solid-state lighting, flat-panel displays but also for sources in novel flexible and wearable applications. This is due to their unique properties, including high efficiency, good color quality, color selectivity and low manufacturing costs through implementation of roll-to-roll processes. OLED devices emitting red light are cutting edge for biosensing applications. Heavy atom doping is one of the most promising and challenging methods amongst the various ways to control the emissive characteristics. Doping regulates the highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) of the host layer in addition to spin conversion of exciton from singlet to triplet. This allows the energy bandgap to be adjusted to a desired level and thus generates light corresponding to that energy level. Moreover, it can act as an energy barrier between the interfaces to change the flow of carriers. In this study, Bis(2-(3,5-dimethylphenyl)quinoline-C,N)(acetylacetonato)iridium(III), also known as Ir(dmpq)2(acac), was doped in various polymeric host materials such as 4,4'-Bis(N-carbazoyl)-1,1'-biphenyl (CBP), 1,3-Bis(N-carbazoyl)benzene (mCP), 1,1-Bis[(di-4-tolylamino) phenyl]cyclohexane (TAPC) and tris(4-carbazoyl-9-ylphenyl)amine (TCTA). The produced films were characterized in terms of their optical properties using Spectroscopic Ellipsometry (SE) and photophysical properties using Photoluminescence (PL) Spectroscopy. Their final evaluation was derived by their implementation in solution processed OLED devices, which were characterized using Electroluminescence (EL) Spectroscopy. We focused on their color purity (Figure 1) and selectivity and significant comparative results were obtained concerning their functionality and performances.

**Keywords:** red OLEDs, Doping, Ir(dmpq)2(acac), Ellipsometry, Photoluminescence, Electroluminescence, 4,4'-Bis(N-carbazoyl)-1,1'-biphenyl (CBP), 1,3-Bis(N-carbazoyl)benzene (mCP), 1,1-Bis[(di-4-tolylamino)

phenyl]cyclohexane (TAPC), tris(4-carbazoyl-9-ylphenyl)amine (TCTA)



**Figure 1:** The CIE color coordinates relative to the EL spectra of the OLED devices with Ir(dmpq)2(acac) doped emissive layers. In the inset, the photograph of the OLED device with CBP host material.

## References:

1. Zhang, Y., Qiao, J. (2021), Near-infrared emitting iridium complexes: Molecular design, photophysical properties, and related applications, *iScience*, 24, 102858.
2. Bansal, A.K., Hou, S., Kulyk, O., Bowman, E.M., Samuel, D.W. (2015), Wearable Organic Optoelectronic Sensors for Medicine, *Adv. Mater.*, 27 7638–7644.

# Synthesis of freestanding porous alumina and metallic nanowires

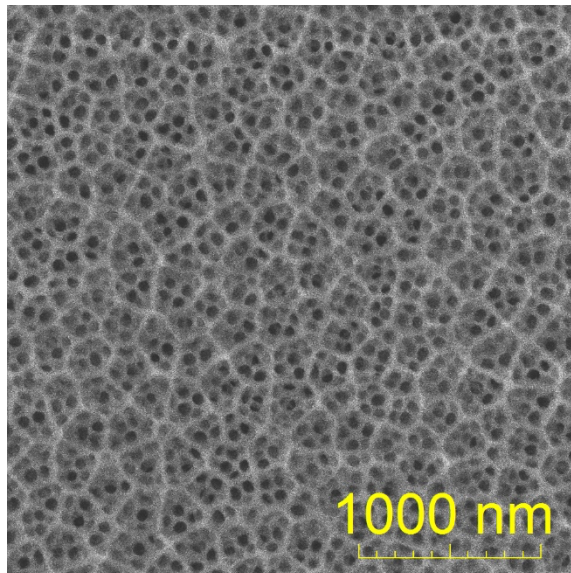
S. Ionescu<sup>1</sup>, C. Gheorghiu<sup>1</sup>, D. Popa<sup>1</sup>, V. Leca<sup>1</sup>

<sup>1</sup> Extreme Light Infrastructure – Nuclear Physics (ELI-NP), Target Laboratory, Horia Hulubei  
National Institute for Physics and Nuclear Engineering (IFIN-HH), Magurele, Romania

## Abstract:

Freestanding porous alumina with micrometer size thickness, along with copper nanowires on aluminium substrate and on gold foil were synthesized by electrochemical methods, to be used as targets for high power laser experiments. The as-received aluminum plates were treated to reduce surface roughness to tens of nanometers, by mechanical polishing, and to enhance metallic grain size, by thermal treatments, prior to the anodization process. To grow the porous alumina (Figure 1) with different characteristics (thickness, pore diameter, interpore distance), process key-parameters (such as: temperature, voltage, electrolyte solution) were adjusted and optimized. After the anodization process, the free-standing alumina, obtained by detaching it from the aluminum substrate by applying stair like reverse biases [1,2], has a thickness between 7-100  $\mu\text{m}$  range, with pore diameter of 30 to 200 nm, 30-100 nm interpore distance and a density of 2-2.7  $\text{g/cm}^3$ . Synthesis of copper and nickel nanowires by means of two routes is also presented: direct current electrodeposition on freestanding alumina and alternative current pulsed electrodeposition on the alumina grown on the aluminum substrate. In each step of the fabrication process the samples were characterized using several surface characterization methods and elemental composition techniques, such as: Scanning Electron Microscopy, Electron Backscatter Diffraction, Atomic Force Microscopy, optical profilometry, optical microscopy, and Energy Dispersive Spectroscopy. The free-standing alumina with open pores on one side or on both sides can be used as targets together with metallic nanowires for high power laser experiments, to increase laser absorption, energy conversion, and the maximum resulted particle energy, as seen from Particle-in-Cell simulations and previous experiments [3].

**Keywords:** freestanding alumina, anodization, nanowires, electrodeposition, laser targets



**Figure 1:** SEM image of porous aluminum oxide with open pores on one side

## References:

1. Hong YK, Kim BH, Kim D Il, Park DH, Joo J. (2015), High-yield and environment-minded fabrication of nanoporous anodic aluminum oxide templates, *RSC Adv* 5:26872–26877
2. Gheorghiu CC, Ionescu SC, Ghenuche P, Cernaianu MO, Doria D, Popa D, Leca V (2021), Structuring Free-Standing Foils for Laser-Driven Particle Acceleration Experiments, *Frontiers in Physics* 9:727498
3. Vallières S, Salvadori M, Permogorov A, Cantono G, Svendsen K, Chen Z, Sun S, Consoli F, d'Humières E, Wahlström C-G, et al. (2021) Enhanced laser-driven proton acceleration using nanowire targets. *Sci Rep* 11:2226.



# Tunable thermal and photochemical crosslinking of CHic-able diazo-groups containing polymers

D. Rusitov<sup>1,2,\*</sup>, J. R  he<sup>1,2</sup>

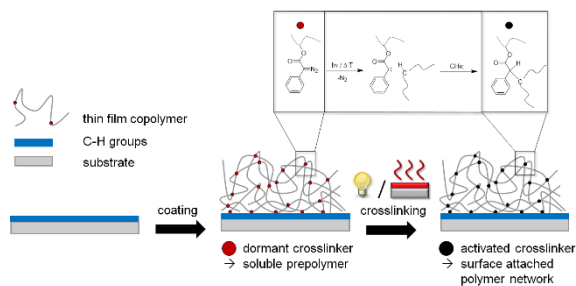
<sup>1</sup> Chemistry and Physics of Interfaces, IMTEK – Department of Microsystem Engineering, University of Freiburg, Freiburg, Germany

<sup>2</sup> Cluster of Excellence *livMatS*, University of Freiburg, Freiburg, Germany

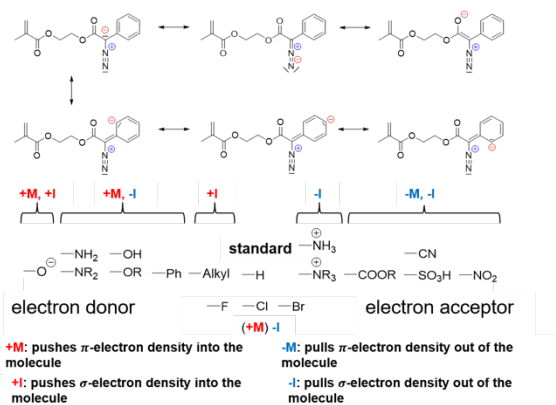
## Abstract:

Crosslinking of polymer networks and their attachment to surfaces is a powerful tool to modify the interaction between a material and the environment. Many methods to achieve polymer networks include crosslinking during polymerization or the use of external reagents. Some disadvantages, however, are that such materials cannot be processed and that reagents may remain in the network, which can diffuse into the environment. A versatile way to overcome these challenges is the C,H insertion based crosslinking (CHic) reaction. A few dormant crosslinker molecules are integrated in a polymer which form reactive groups through UV or thermal activation and react with any C,H group in direct vicinity to form a network. Simultaneously surface attachment occurs when the surface has C,H bonds (Figure 1). Reactive intermediates, which undergo this type of reaction, are ketyl biradicals, nitrenes or carbenes. One way to generate carbene intermediates is upon thermal or photochemical activation of diazo-groups. This activation is controllable to a certain extend through the chemical environment of the diazo group. With the use of acceptor or donor groups, it is possible to tune the thermal activation parameter through stabilization or destabilization of the diazo group (Figure 2). The photochemical behaviour is controllable using the  $\pi$ -electron system to go from UV-activation to activation via visible light. Through the control of the diazo decomposition, it is possible to tune the crosslinker activation parameter to the desired application.

**Keywords:** polymer networks, crosslinking, CHic, diazo, carbene, gels, surface attachment, thermal/photochemical activation, chemical tuning, donor/acceptor groups



**Figure 1:** Figure illustrating the CHic reaction of diazo groups to form surface attached polymer networks. Polymers can be processed and coated and then in situ crosslinked via thermal or photochemical activation.



**Figure 2:** Figure illustrating the electronic structure of a phenylester diazo crosslinker (PEDAz). Donor groups on the aromatic ring facilitate diazo decomposition because the molecule is destabilized due to too much negative electronic charge density. Acceptor groups therefore stabilize diazo decomposition.

## References:

- O. Prucker, T. Brandstetter, J. R  he, *Biointerphases* 13, 010801 (2016).
- J. Kost, A. Bleiziffer, D. Rusitov, J. R  he, *Journal of the American Chemical Society* 2021, 143, 10108.

## Dual-phase CuZr thin film metallic glasses (TFMGs) deposited by PVD magnetron sputtering: bias voltage and thickness dependency of structure, hardness, and wear resistance

Alireza Bagherpour <sup>a\*</sup>, Marie-Stéphane Colla <sup>b</sup>, Andrey Orekhov <sup>c</sup>, Thomas Pardoën <sup>b</sup>, Stéphane Lucas <sup>a</sup>

<sup>a</sup> Laboratoire d'Analyse par Réaction Nucléaires (LARN), Namur Institute of Structured Matter (NISM), University of Namur, 61 Rue de Bruxelles, 5000 Namur, Belgium

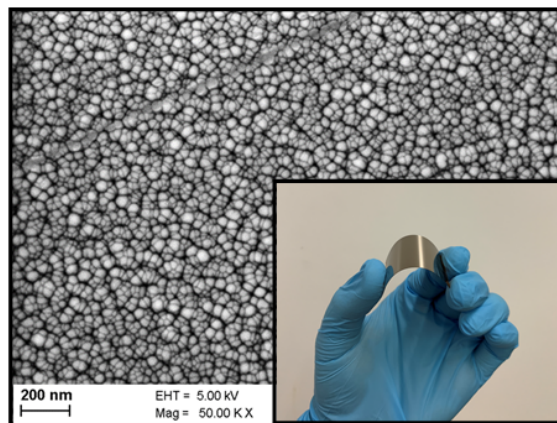
<sup>b</sup> Institute of Mechanics, Materials and Civil Engineering (iMMC), UCLouvain, Place Sainte Barbe 2, bte L502.02, B-1348, Louvain-la-Neuve, Belgium

<sup>c</sup> EMAT, University of Antwerp, Groenenborgerlaan 171, B-2020, Antwerp, Belgium

### Abstract:

Bulk metallic glasses (BMGs) show appealing mechanical properties including high hardness, desirable corrosion resistivity, and thermal characteristics[1],[2]. BMGs suffer from brittleness due to their short-range structure and lack of the dislocation-like crystalline defects. The thin film configuration (TFMGs) has received a lot of attraction in recent decades due to additional interesting properties as high strength, ductility sometimes beyond 10%, and notable corrosion and wear resistance[3],[4]. In this study, the structure of crystalline Cu/Zr and novel multilayered dual-phase CuZr TFMGs deposited by PVD magnetron sputtering are compared for different deposition conditions (0V, 100V, 150V, 200V bias voltage) and various coating thicknesses (230nm, 420nm, 620nm, 700nm). Films characterization has been done by XRD, SEM, TEM, XPS, and Profilometer. Hardness and wear resistance relative to deposition conditions were studied by nano-indentation and pin-on-disc respectively. Improved mechanical properties are observed in dual-phase CuZr TFMG sample in comparison with crystalline Cu/Zr and mono-phase CuZr films. According to our observation, increasing bias voltage act as a double edge sword: it leads to an increase in mechanical properties then decreases above 150V. Higher thickness leads to a decline in mechanical properties. Overall, the best mechanical properties were achieved for 150V bias and below 500nm dual-phase metallic glass thin film.

**Keywords:** Metallic glasses, Thin films, Cu-Zr alloys, Magnetron sputtering.



**Figure 1:** Hard enough Flexible CuZr TFMG with columnar nano-structure.

### References:

1. J. P. Chu *et al.*, "Thin film metallic glasses: Unique properties and potential applications," *Thin Solid Films*, vol. 520, no. 16, pp. 5097–5122, 2012.
2. M. F. Ashby and A. L. Greer, "Metallic glasses as structural materials," *Scr. Mater.*, vol. 54, no. 3, pp. 321–326, Feb. 2006.
3. M. Ghidelli, S. Gravier, J. J. Blandin, T. Pardoën, J. P. Raskin, and F. Mompiau, "Compositional-induced structural change in  $Zr_{x}Ni_{100-x}$  thin film metallic glasses," *J. Alloys Compd.*, vol. 615, no. S1, pp. S348–S351, Jan. 2015.
4. M. Apreutesei, P. Steyer, A. Billard, L. Joly-Pottuz, and C. Esnouf, "Zr-Cu thin film metallic glasses: An assessment of the thermal stability and phases' transformation mechanisms," *J. Alloys Compd.*, vol. 619, pp. 284–292, 2015.

# Post Processed Ceramic Based Coatings on Steels using Thermal Spray

A. Pattnayak<sup>1</sup>, A. Gupta<sup>1</sup>, N.V. Abhijith<sup>1</sup>, D. Kumar<sup>1\*</sup>, J. Jain<sup>2</sup>, V. Chaudhry<sup>3</sup>

<sup>1</sup> Centre for Automotive Research and Tribology, Indian Institute of Technology Delhi, New Delhi, India

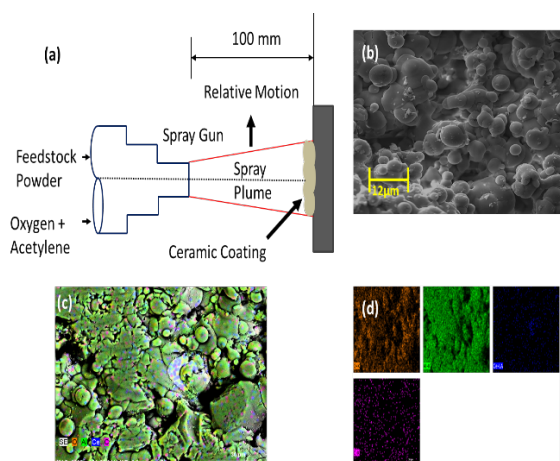
<sup>2</sup> Department of Material Science and Engineering, Indian Institute of Technology Delhi, New Delhi, India

<sup>3</sup> Nuclear Power Corporation of India Limited, Mumbai, Maharashtra, India

## Abstract:

Hard and dense ceramic coatings deposited on the surface through conventional powder flame spray (CPFS) process provide ideal solution to the poor tribological properties exhibited by some popular stainless steels like EN-36, 17-4PH etc. These steels are widely used in nuclear, fertilizer, food processing, marine industries under extreme environmental conditions. This CPFS is generally accompanied by post fusion processes like laser treatment, inductive heating, oxyacetylene flame heating etc. Post fused coatings exhibit different properties than the as-sprayed ones. The present study focuses on the comparative study of the properties of As-sprayed and post processed  $\text{Al}_2\text{O}_3$ -0.8 CeO<sub>2</sub>-0.2 rGO coatings deposited on 17-4PH steel using CPFS. The post fusion is done by oxyacetylene flame. Figure 1 shows the microstructure and elemental mapping of As Sprayed coating. Further, we report the physical (density, surface roughness, hydrophobicity), Metallurgical (SEM, XRD, Raman), Mechanical (Hardness), Tribological (wear, scratch hardness, corrosion) characterization of both As-sprayed and post processed coatings. The comparison of the properties will help us to reveal the necessity and challenges of post fusion of such coatings.

**Keywords:** thermal spray process, CPFS, ceramic coating, hardness, oxyacetylene heating, post fusion



**Figure 1:** Figure illustrating (a) Schematic representation of CPFS process, (b) SEM micrograph of  $\text{Al}_2\text{O}_3$ -0.8 CeO<sub>2</sub>-0.2 rGO As-sprayed coatings and (c) and (d) are EDS and elemental mapping of the same coating.

## References:

1. V. Chaudhry, S. V. Kailas (2013), Fretting studies on self-mated stainless steel and chromium carbide coated surfaces under controlled environment conditions, *Wear*. 301 524–539.
2. G.J. Li, J. Wang, C. Li, Q. Peng, J. Gao, B.L. Shen (2008), Microstructure and dry-sliding wear properties of DC plasma nitrided 17-4 PH stainless steel, *Nucl. Instruments Methods Phys. Res. Sect. B Beam Interact. with Mater. Atoms*. 266 1964–1970.



## Effects of 18 Ni (300) maraging steel coating by laser deposition on LPBF AISI 316L component

V. Errico<sup>1,\*</sup>, P. Posa<sup>1</sup>, A. Angelastro<sup>1</sup>, M. Mazzarisi<sup>1</sup>, A. Fusco<sup>1</sup>, S.L. Campanelli<sup>1</sup>

<sup>1</sup>Department of Mechanics, Mathematics, and Management, Politecnico di Bari, Bari, Italy

### Abstract:

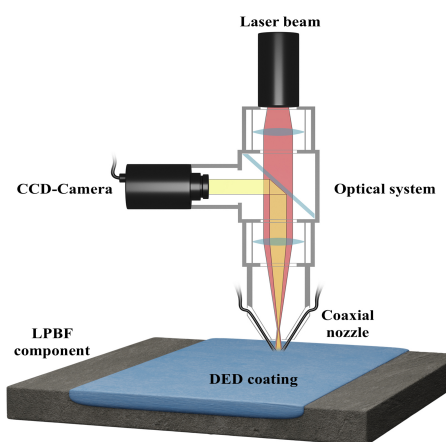
As a result of Additive Manufacturing (AM) processes it is often possible to obtain mechanical parts presenting poor surface quality due to an unacceptable surface roughness or a weak metallurgical structure. Looking for a solution among laser-based and additive manufacturing processes, very interesting answers can be found in the laser remelting, laser cladding and directed energy deposition processes. These solutions have the capability of improving the surface properties in agreement to the design requirements and modifying the surface shape by adding material for reconfiguration, restoration, or repairing operations.

This study was carried out on AISI 316L components made by using the Laser Powder Bed Fusion (LPBF) technology, which is a process getting a growing interest from an academic and industrial point of view. By means of LPBF is possible to build parts having very complex geometries, allowing a high freedom of design. At the same time, it is possible to reduce the time to market and realize a more sustainable manufacturing for small productions volumes. However, the large-scale deployment of this process has been hindered by technical challenges that still have to be overcome.

In order to fix these issues and explore the capabilities of the laser treatments supported by powder injection, a Directed Energy Deposition (DED) coating, using 18 Ni (300) maraging steel powder (Figure 1), and a subsequent heat treatment were performed. The results revealed the goodness of the coating and the improvement of the surface properties in the LPBF component. The microstructure at the interface between the coating and the component was also analyzed. Through the support of a coaxial optical monitoring system, which enabled a real-time monitoring of the process, a stable treatment was obtained.

This study has several possible applications, especially in manufacturing complex molds or tools, even with internal cooling channels, which can be coated in order to achieve enhanced surface properties compared to the core, in terms of wear resistance and hardness.

**Keywords:** Laser Powder Bed Fusion, 18 Ni (300) maraging steel, Directed Energy Deposition, Optical Process Monitoring, Heat Treatment.



**Figure 1.**

Schematic overview of the Directed Energy Deposition system used for coating, equipped with the optical setup deployed for process monitoring and control.

### References:

1. Feenstra, D.R., Banerjee, R., Fraser, H.L., Huang, A., Molotnikov, A., Birbilis, N. (2021), Critical review of the state of the art in multi-material fabrication via directed energy deposition, *Curr. Opin. Solid State Mater. Sci.*, 25,100924.
2. Ben-Artzy, A., Reichardt, A., Borgonia, J.P., Dillon, R.P., McEnerney, B., Shapiro, A.A., Hosemann, P. (2021), Compositionally graded SS316 to C300 Maraging steel using additive manufacturing, *Mater. Des.*, 201,109500.
3. Errico, V., Fusco, A., Campanelli, S.L. (2022), Effect of DED coating and DED + Laser scanning on surface performance of L-PBF stainless steel parts, *Surf. Coatings Technol.*, 429,127965.

# Properties of coatings and SPS sinters made of tungsten diboride alloyed with Ti, Cr, Mo, Re and Zr

R. Psiuk<sup>1,\*</sup>, M. Wiśniewska<sup>2</sup>, D. Garbiec<sup>2</sup>, T. Mościcki<sup>1</sup>

<sup>1</sup> Institute of Fundamental Technological Research PAS, Warsaw, Poland

<sup>2</sup> Research Network Łukasiewicz Metal Forming Institute, Poznań, Poland

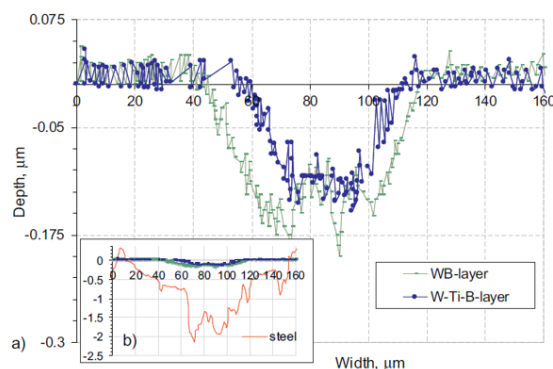
## Abstract:

Tungsten borides due to their properties can be an alternative to superhard materials. While they possess high hardness, chemical and thermal resistance, they are easier to manufacture than diamond and cubic boron nitride. Tungsten borides in bulk form can be produced without employing high pressures (>5GPa), and in case of coatings there is no need to use processes with high plasma density. Tungsten boride properties can be improved by alloying with various transition metals, e.g. titanium (Figure 1). To enhance the properties of tungsten diboride (WB<sub>2</sub>), we have synthesized and characterized solid solutions of this material with titanium, chromium, molybdenum, rhenium and zirconium. The obtained materials were subsequently deposited as a coatings. Various concentrations of these transition-metal (TM) elements, ranging from 0.0 to 24.0 at. %, on a metals basis, were made. Spark plasma sintering (SPS) was used to synthesize these refractory compounds from the pure elements. Elemental and phase purity of the both samples (sinters and coatings) were examined using energy-dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD), and microindentation was utilized to measure the Vickers hardness under applied loads of 200 gf. XRD results indicate that the solubility limit is below 8 at. % for Mo, Re, Zr, and below 16 at. % for Cr. Above this limit both diborides (W,TM)B<sub>2</sub> are created. Addition of TM caused decrease of density and increase of hardness and electrical conductivity of sinters. Deposited coatings W<sub>1-x</sub>TM<sub>x</sub>B<sub>2-z</sub> (x=0.24, z=0.2—0.07) are homogenous, smooth, and hard. Coatings with addition of Mo, and Zr have hardness above 40 GPa. Addition of Cr increased the films hardness to 50 GPa. Coatings alloyed with Zr exhibits fracture toughness values of 2.11 MPa·m<sup>1/2</sup>, which is value similar to TiN or CrN/TiN coatings.

**Keywords:** tungsten boride, superhard materials, spark plasma sintering SPS, magnetron sputtering

**Acknowledgement** This work was funded by the National Centre for Research and Development

(NCBR, Poland) under project No. TECHMATSTRATEGIII/0017/2019.



**Figure 1:** Figure illustrating the increase of wear resistance of tungsten diboride alloyed with titanium.

## References:

1. Mościcki T., Psiuk R., Słomińska H., Levintant-Zayonts N., Garbiec D., Pisarek M., Bazarzik P., Nosewicz S. (2020) Influence of overstoichiometric boron and titanium addition on the properties of RF magnetron sputtered tungsten borides, *Surf. and Coat. Tech.*

# The Influence of Ambient Cure Chemistry and Stoichiometry on the Surface Properties of Epoxy Coatings for Industrial Application

C. Bannister<sup>1</sup>, A. Guy<sup>2</sup>, R. Thompson<sup>3</sup>

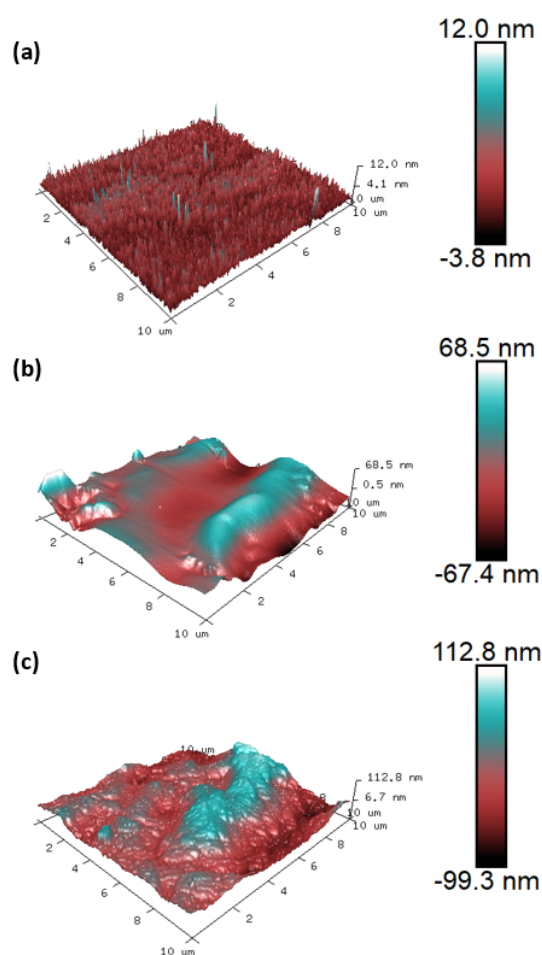
<sup>1,3</sup> Department of Chemistry, Durham University, Durham, United Kingdom

<sup>2</sup> Safinah Ltd, Gateshead, United Kingdom

## Abstract:

During industrial application of multicoat epoxy based paint systems, ambient cure conditions dictating first coat properties are often varied, the impact of which are not fully understood or characterized. Epoxy-epoxy interfacial adhesion is hypothesised to be influenced by first coat surface properties (as is consistent with interfacial chemistry<sup>1</sup>), so thorough first coat surface characterisation is needed to understand adhesion failure in two coat industrial epoxy systems. Variation in ambient cure conditions (temperature and relative humidity), stoichiometry and delay time between epoxy component mixing and film casting ("induction time") significantly altered the surface properties of ambiently cured epoxy resin coatings (DEN-431, RDGE and PAC-M). Gravimetric analysis shows that induction time significantly reduced surface layer formation (carbamation) of cured epoxy resin coatings at elevated relative humidity (80%) but had no effect at lower humidity (40%). RMS surface roughness increases with increasing relative humidity and decreases with increasing induction time and ambient cure temperature, at two stoichiometric extremes. However the net change in surface area arising from these conditions was not sufficient to significantly alter the equilibrium contact angles or wetting regime. We therefore conclude that the observed significant variation in surface wettability was more likely to depend on variation in surface chemistry than roughness. Onset ambient cure system T<sub>g</sub> was unaffected by variation in relative humidity at both stoichiometric extremes but increasing ambient cure temperature significantly increased off-stoichiometry onset ambient cure system T<sub>g</sub>.

**Keywords:** epoxy, epoxy resin, polymer, coating, cure, cure conditions, stoichiometry



**Figure 1:** AFM 3D Images of on stoichiometry DEN 431-RDGE-PAC-M films cured at (a) 0% RH (b) 40% RH (c) 80% RH. In each case, vertical scales have been optimised to highlight any variation in surface height.

## References:

1. A. Karim and S. Kumar, *Polymer Surfaces, Interfaces and Thin Films*, World Scientific, 2000.

# Study of bioinspired super-hydrophobic polymer textured surfaces to design fluorine-free high repellency textiles

Q. Legrand<sup>1</sup>, S. Benayoun<sup>1</sup>, S. Valette<sup>1\*</sup>

<sup>1</sup> Laboratory of Tribology and Systems Dynamics, Ecole Centrale de Lyon, France

\*stephane.valette@ec-lyon.fr

## Abstract:

Due to sanitary and environmental considerations, fluorine-free approaches have to be developed to maintain super-hydrophobic properties of textiles at the horizon of 2030 in Europe. A dual approach based on physical texturing and fluorine-free chemistry is proposed.

Physical surface texturing is based on three points:

- The determination of morphological and topographical parameters from models of the living world through a bio-inspired approach;
- The implementation of a numerical code of analytical modeling of the wetting of model textured surfaces as a tool to help the design of super-hydrophobic patterns;
- The fine experimental study of the static and dynamic wettability properties of textured model surfaces and the comparison with numerical models.

Three plant surfaces are studied in the context of a bio-inspired approach. The quantitative description of the surface topographic parameters allows, through a frequency filtering approach, to decompose the surface topography in several scales. In order to separate the chemical component from the topographic component, a PDMS replication method is implemented. This method allows the replication of the micrometric topographies and not the nanometric one. Due to the micrometric scales, an increase of about 20° is shown for the static contact angles, while at the same time the contact angle hysteresis decreases from 40° to about 10° after texturing [1].

The micrometric topographic parameters resulting from the biomimetic analysis are then incorporated into an analytical wetting modeling code based on the Extrand's approach [2]. This code allows to model the wettability behaviors of Wenzel and Cassie-Baxter modes. Wetting transitions between these two modes can be predicted as a function of the topographic parameters modeled.

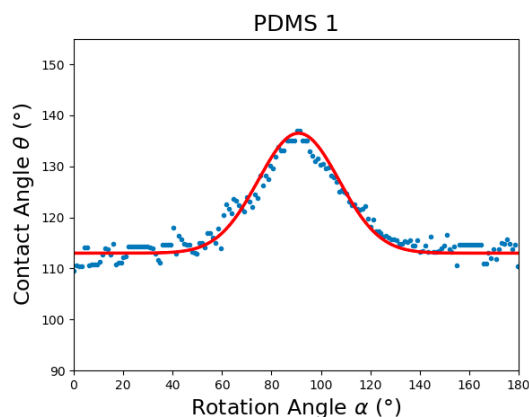
Finally, the experimental wettability results confirm the importance of the multiscale aspects in the wettability of textured surfaces as well as the predictions of the wetting transitions obtained

with the analytical code. Moreover, the anisotropic spreading on textured surfaces is also confirmed through 3D-goniometer wetting analyses (Figure 1).

The coupling of the experimental tests with the results of the analytical wetting modeling provides a new tool for the design of super-hydrophobic topographic patterns.

Coupled with the development of new molecules, the topographic surface patterns obtained become good candidates for water repellency of fluorine-free textiles.

**Keywords:** super-hydrophobicity, surface texturing, biomimetics, wetting, fluorine-free textile, polymer, femtosecond laser, PDMS replication, analytical modeling.



**Figure 1:** Experimental evolution of the static contact angle ( $\theta$ ) as a function of the rotation angle ( $\alpha$ ). The gaussian-like evolution is modelled through both experimental and mathematical considerations of the droplet anisotropic spreading.

## References:

1. Legrand Q., Benayoun S., Valette S. (2021), Biomimetic approach for the elaboration of highly hydrophobic surfaces: study of the links between morphology and wettability, *Biomimetics*, 6, 38.
2. Extrand C. W. (2002), Model for contact angles and hysteresis on rough and ultraphobic surfaces, *Langmuir*, 18, 7991-7999.

## Development and investigation of textile materials with Conductive Coatings intended for microwave shielding and absorption

V. Rubežienė<sup>1,\*</sup>, A. Sankauskaitė<sup>2</sup>, S. Varnaitė-Žuravliova<sup>1</sup>, A. Abraitienė<sup>2</sup>

<sup>1</sup> Department of Textiles Physical-Chemical Testing, Center for Physical Sciences and Technology, Kaunas, Lithuania

<sup>2</sup> Department of Textile Technologies, Center for Physical Sciences and Technology, Kaunas, Lithuania

### Abstract:

The reduction of electromagnetic radiation (EMR) impact is very important for the protection of people frequently using electrical and electronic devices which can emit electromagnetic waves with frequencies that are potential hazards to health. The most utilized range is the microwave range, which can be defined as 1 - 40 GHz, as most of the modern point to point, wireless, and satellite communications occupy this range. Electrically conductive fabrics with particular EMR shielding properties not only offer an opportunity to counter these threats, but also can be applicable to develop radar absorbing materials (RAM), for use in the field of stealth technology to disguise a vehicle or soldier from radar detection.

In this study the results of development and investigation on different textile fabrics coated with conductive coatings are presented. Textile materials with incorporated conductive additives or coated with special conductive formulations are electrically conductive and therefore interact with the electric component of electromagnetic waves (EMR). Among EMR shielding textile materials only materials with substantial contribution to shielding from absorption have the potential to be used as RAM. To develop the fabrics with microwave shielding and absorbing properties samples of woven and knitted fabrics were coated with compositions containing inherently conducting polymers (ICPs), carbon-based formulations or their mixtures. For coating conventional textile coating technologies - screen printing and knife-over-roll, were applied. The investigation of reflection and transmission properties of developed textile fabrics was performed in a frequency range of 2–18 GHz.

It was found that shielding effectiveness (SE) as well as absorption properties depend not only on the amount and type of conductive formulation topped on the fabric, but also resides in the construction parameters of fabrics and their finishing before coating. Depending on such fabric structural parameters as—density, mass per unit area, type of weave, layer of shield (or coating) just

sticks on the fabric surface or penetrates into fabric changing the shield thickness herewith turning SE results. Meanwhile, the fiber composition of fabrics mostly influences the bonding between fibers and coating. By controlling coating deposit on the fabric, it is possible to tune the electrical properties to a certain extent and hereby influence the reflection and transmission parameters of the coated textile material. The type of coating technology also influences the electromagnetic parameters of fabrics coated with the same composition.

This research has been carried out within the Project ACAMSII, “Adaptive camouflage for the soldier”, which has received funding from the European Union’s Preparatory Action for Defence Research (PADR) programme under grant agreement No 800871.

**Keywords:** electromagnetic radiation, microwave range, shielding, coating, deposit, reflection, transmission, textile, inherently conducting polymers

# OXIDATION KINETICS OF INCONEL 625® UNDER CO<sub>2</sub>

Boris CONTRI<sup>1</sup>, Stéphane VALETTE<sup>1</sup>, Marina SOUSTRE<sup>1</sup>, Pierre LEFORT<sup>1</sup>  
 IRCER (European Ceramics Center), UMR CNRS 7315, University of Limoges,  
 12, rue Atlantis 87068 Limoges Cedex, France

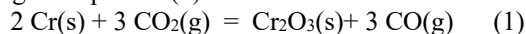
## Abstract:

Nickel-chrome based alloy Inconel 625® is widely used in high temperature environments. Its high good behavior at high temperature [1] allows its usage in many industrial devices, such as thermal exchangers.

Many papers [2-3] were devoted to the behavior of Inconel 625® in air, oxygen and sulfur dioxide but none in CO<sub>2</sub> which represent a significant part of the industrial gas environment encountered such as in the waste to energy plants. The present study focused on this topic, mainly in order to understand the first stages of this alloy oxidation in CO<sub>2</sub>.

A common quality Inconel 625® has been used. The oxidation isotherms, followed between 1173 and 1273 K in flowing CO<sub>2</sub> at atmospheric pressure, exhibited parabolic shapes, associated to an activation energy of  $310 \pm 18$  kJ.mol<sup>-1</sup>. Concerning the influence of CO<sub>2</sub> pressure, it was shown that there was not any influence on the reaction rate, up to pressures as low as 0.01 Pa.

The main phase formed was chromia Cr<sub>2</sub>O<sub>3</sub>, with XRD pics shifted, which indicated the presence of traces of other alloy elements inside its structure. On this basis, the reaction considered was simplified following the equation (1):



The reaction mechanism proposed implies the dissociative sorption of carbon dioxide at the external interface, the outer growth of the oxide layer, accompanied by the chrome oxidation at the internal interface following the equation (2) (using the Kröger-Vinck formalism):

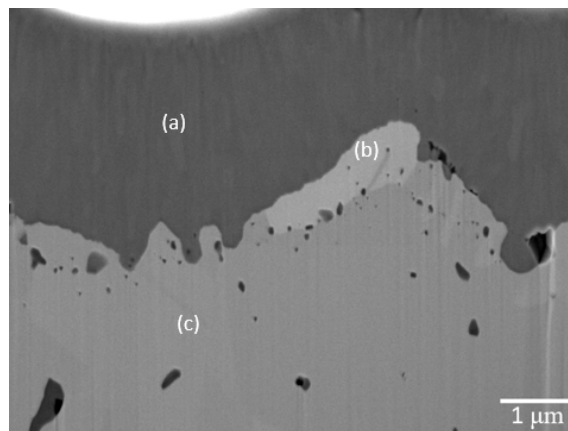


and, lastly, the centrifugal diffusion of chromium atoms in Inconel 625® (i.e. the centripetal diffusion of the chromium vacancies).

It was proved that this last step of vacancies diffusion inside the alloys was the limiting step of the oxidation reaction.

This result is not in agreement with what is generally admitted in the case of the oxidation of chromia forming alloys, in oxygen or air as well as in CO<sub>2</sub>. Indeed, it is usually considered that the stainless steel alloys are passivated by the chromia oxide layer, while, in the present study, it was demonstrated that this layer played not the main protection role for the alloy.

**Keywords:** Inconel 625®, CO<sub>2</sub>, Kinetics, Oxidation, FIB-SEM



**Figure 1:** Cross-section of Inconel 625® oxidized at 1223 K, with (a) chromia layer, (b) Nb-phase, (c) Inconel 625®

## References:

1. Lewis E. Shoemaker, « Alloy 625 and 725: Trends in properties and applications », Superalloys 718, 625, 706 and Derivatives, TMS (2005)
2. Petrzak, P., K. Kowalski, et M. Blicharski. « Analysis of Phase Transformations in Inconel 625 Alloy during Annealing ». Acta Phys. Pol. 130, no 4 (2016): 1041-44.
3. F. Tehovnik, J. Burja, S. Malej, F. Vode, B. Podgornik, B. Arh, B. SetinaBatic, « Evolution of microstructure during Hot deformation of Inconel 625 alloy with different strain rates» Metalurgija 58 (2019).



# Evaluation and improvement of adhesion between polymeric nanofibers and metallic substrates

T. Schneiders<sup>1\*</sup>, F. Ahrens<sup>2</sup>, T. Gries<sup>1</sup>

<sup>1</sup> Institut fuer Textiltechnik, RWTH Aachen University, Germany

<sup>2</sup> Institute of Biotechnology, RWTH Aachen University, Germany

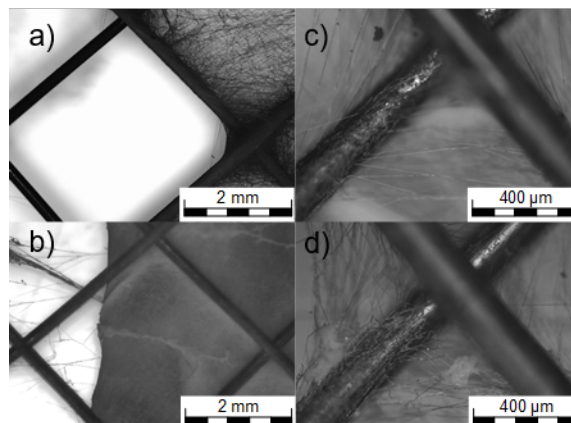
## Abstract:

Polymeric nanofibers have promising properties for medical applications and Tissue Engineering. These are high specific surface area, adjustable pore size, and scale of extracellular matrix. However, they lack mechanical strength and stability, therefore often need a substrate for bearing the load and giving stability. In medical applications, this is often a metallic material like a stent or metallic surface of an implant. The integration of nanofibers and the adhesion between the two structures has not been sufficiently researched until today. Therefore, this study aims to find a method to evaluate the adhesion between a metal substrate and polymeric nanofibers from electrospinning and develop methods for improving the adhesion to a sufficient level.

Nanofibers from thermoplastic polyurethane (TPU) were produced using the solution electrospinning method. Electrostatic forces are used to draw fibers with diameters on the nanometer scale from a polymer solution and produce nonwovens out of it in this technology. The nanofibers were directly spun on a cleaned and prepared Nitinol plate. The samples were tested in a 180° peel test according to DIN EN ISO 11339 and mean peel forces calculated from the stress-strain curves. The results for bare Nitinol plates were set as the baseline. Additionally, methods for adhesion and bonding improvement were tested. Heat treatment and solvent vapor treatment as a post-treatment, film coating with TPU, and argon plasma treatment as a pretreatment were used.

All methods were able to increase peel forces significantly. The untreated coated samples have a peel force/width of  $99.8 \pm 29.5$  mN/cm and the highest measured peel force/width appears for the precoated samples of  $556.6 \pm 211.7$  mN/cm. Hence, the maximal increase of peel force/width in this study was around 5.5 times.

**Keywords:** electrospinning, nanofibers, Nitinol, stent coating, adhesion, plasma treatment, peel force.



**Figure 1:** Nanofiber covered Nitinol struts from the uncoated side. a) and b) show halfway peeled of nanofiber coatings. In a) the nanofiber coating is sticking to the struts and in b) it is not. c) and d) are upclose pictures of the Nitinol struts, showing the nanofibers wrapped around the struts. This increases the adhesion due to form fit and bonding compared to the adhesion between a plate and a nanofiber coating.

## References:

1. Ballarin, F.M., Blackledge, T.A., Capitos Davis, N.L., Frontini, P.M., Abraham, G.A. and Wong, S.-C. (2013), Effect of topology on the adhesive forces between electrospun polymer fibers using a T-peel test. *Polym Eng Sci*, 53, 2219-2227.
2. Hellert C, Wortmann M, Frese N, Grötsch G, Cornelißen C, Ehrmann A. Adhesion of Electrospun Poly(acrylonitrile) Nanofibers on Conductive and Isolating Foil Substrates. *Coatings*. 2021; 11(2):249
3. Zhang, B., Yan, X., Xu, Y., Zhao, H., Yu, M., Long, Y. (2020) Measurement of Adhesion of In Situ Electrospun Nanofibers on Different Substrates by a Direct Pulling Method. *Advances in Materials Science and Engineering*, 2020, 8 pages



# Effect of the detonation spraying technological parameter on the phase composition and properties of $\text{Cr}_3\text{C}_2$ - NiCr coatings

N.M.Magazov<sup>1\*</sup>, B.K. Rakhadilov<sup>2</sup>, D.N. Kakimzhanov<sup>1,2</sup>, L.G. Zhurero<sup>3</sup>

<sup>1</sup>D. Serikbayev East Kazakhstan technical university, Ust-Kamenogorsk, Kazakhstan

<sup>2</sup>PlasmaScience LLP, Ust-Kamenogorsk, Kazakhstan

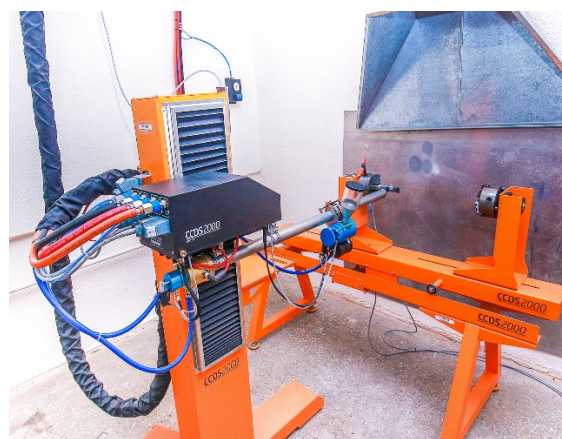
<sup>3</sup>Sarsen Amanzholov East Kazakhstan University, Ust-Kamenogorsk, Kazakhstan

## Abstract:

The application of protective coatings to improve the mechanical, physicochemical and tribotechnical properties of metals and alloys and products made from them is an urgent field of materials science, solid-state physics, plasma physics, chemistry etc.  $\text{Cr}_3\text{C}_2$ -NiCr coatings are widely used to protect against the wear of working bodies at high temperatures. The plastic metal matrix NiCr served as a binder supporting the brittle reinforcing phase  $\text{Cr}_3\text{C}_2$ . The paper considers the results of a study of the phase composition and properties of detonation coatings based on  $\text{Cr}_3\text{C}_2$ -NiCr, obtained at different values of the detonation barrel filling volume with an explosive acetylene-oxygen mixture. The detonation barrel filling volume with an explosive gas mixture varied from 51% to 64%. Detonation coatings based on  $\text{Cr}_3\text{C}_2$ -NiCr, at different filling volume values (51%, 58%, 64%) were obtained on a computerized detonation spraying complex of the new generation CCDS2000 (Figure 1). The samples microhardness was measured on the Metolab-502 device. The coatings tribological characteristics were studied on the Anton Paar TRB<sup>3</sup> tribometer by the ball-disc method. The coatings surface roughness was evaluated using Ra's parameter on the profilometer model 130. The samples phase composition was studied by X-ray diffraction analysis on an X'PertPro diffractometer. The study results of the samples phase composition showed that all samples have component phases:  $\text{Cr}_3\text{C}_2$ ,  $\text{Cr}_3\text{O}_4$ ,  $\text{Cr}_2\text{O}_3$  and  $\text{Cr}_{23}\text{C}_6$ , but from the diffractograms, it can be seen that, depending on the barrel filling volume, the phase compositions intensity of the coatings varies. This may be due to the high heating temperature of the powder during spraying since the heating temperature of the powder strongly depends on the barrel filling volume. The tribological tests showed that all coatings have a friction coefficient in the range of 0.06-0.45. The coating obtained when filling the barrel in 58% showed relatively low wear (0.27). This is due to an increase in the volume fraction of the  $\text{Cr}_2\text{O}_3$  phase. It is determined that the surface of all coatings have an inhomogeneous structure with pores, a typical layered, undulating arrangement of structural

components. As the main parameter for measuring surface roughness, the parameter Ra was chosen, which is the arithmetic mean deviation of the profile. The study results of the roughness of the coatings showed that the effect of filling the detonation barrel with an explosive mixture on the roughness is insignificant. The microhardness study results showed that the coating obtained at 58% filling has a high hardness of 1125HV. This may also be due to an increase in the volume fraction of  $\text{Cr}_2\text{O}_3$ , which has good wear resistance.

**Keywords:** detonation spraying, coatings, hardness, roughness, phase composition.



**Figure 1:** Computerized detonation spraying complex of the new generation CCDS2000

## References:

1. Kakimzhanov, D. N., Rakhadilov, B. K., Tyurin, Y. N., Kolisnichenko, O. V., Zhurero, L. G., Dautbekov, M. K. Influence of pulsed plasma treatment on phase composition and hardness of  $\text{Cr}_3\text{C}_2$ -NiCr coatings. *Eurasian Journal of Physics and Functional Materials*, 2021, Vol.5(1), pp. 45-51.
2. Rakhadilov, B., Kakimzhanov, D., Baizhan, D., Muslimanova, G., Pazyzbek, S., Zhurero, L. Comparative study of structures and properties of detonation coatings with  $\alpha$ - $\text{Al}_2\text{O}_3$  and  $\gamma$ - $\text{Al}_2\text{O}_3$  main phases. *Coatings*, 2021 11(12).

# Hybrid nanostructures for (photo)electrochemical water splitting

S. Kment<sup>1,2,\*</sup>, A. Naldoni<sup>1</sup>, R. Zboril<sup>1,2</sup>

<sup>1</sup>Regional Centre of Advanced Technologies and Materials, Czech Advanced Technology and Research Institute, Palacký University Olomouc, Šlechtitelů 27, Olomouc, 783 71, Czech Republic

<sup>2</sup>Nanotechnology Centre, Centre of Energy and Environmental Technologies, VŠB–Technical University of Ostrava, 17. listopadu 2172/15, 708 00 Ostrava-Poruba, Czech Republic

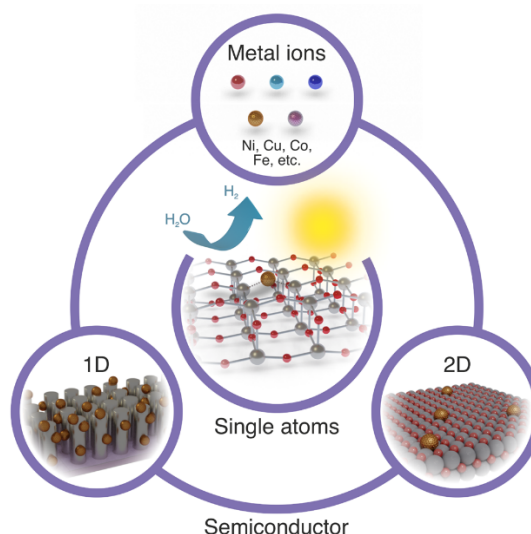
## Abstract:

The production of energy through solar light harvesting and conversion is considered among the most promising options for tackling the growing need for energy on global scale and environmental concerns caused by the huge dependence of the energy demand on fossil fuels. In spite of the social relevance of these energy-related issues, finding valuable alternatives to the use of oil-based energy sources remains highly challenging and thus sustainable technologies are still far from commercial and industrial applications. Semiconductor metal oxides (e.g.,  $\text{TiO}_2$ ,  $\alpha\text{-Fe}_2\text{O}_3$ ,  $\text{BiVO}_4$ ) are the most widely adopted materials for the conversion of solar energy into transportable chemical energy such as di-hydrogen ( $\text{H}_2$ ) produced via the photocatalytic and photo-electrochemical (PEC) splitting of water. Nowadays, the scientific research is mainly focused on the development of effective strategies that could couple (i) light absorption, (ii) steady-state production and transfer of photogenerated charge carriers, and (iii) solar water splitting reaction into one single device, and on the engineering of composite assemblies that could maximize the efficiency of the single processes. Unfortunately, none of the previously mentioned metal oxides simultaneously meet the thermodynamic and kinetic requirements that would ensure a high performance in the mutually interconnected (i) – (iii) processes.

This presentation will deal with the design and realization of hybrid nanostructures, in which selected components are arranged to leverage their expected mechanistic functions. In particular we aim at covering the lack of efficient multifunctional PEC materials and devices by: i) controlling dimensionality (i.e., 1D - 3D) for improved charge transport directionality; ii) engineering hybrid nanostructures (HNS), that could provide enhanced water splitting efficiency through the synergistic combination of the single semiconductor counterparts' properties; iii) selecting novel co-catalysts/sensitizers, to boost charge transfer processes and to extend light absorption properties

## Keywords

Hydrogen, Water splitting, Photoactive semiconductors, Hybrid nanostructures, single atom co-catalysts



**Figure 1:** The presentation describes a strategy how to incorporate single atoms co-catalysts into photoactive semiconductors (1D and 2D morphologies) based on controllable defect nano-engineering.

## References:

1. Kment, S., Riboni, F., Pausova, S., Wang, L., Han, H., Hubicka, Z., Krysa, J., Schmuki, P., Zboril, R. (2017) Photoanodes based on  $\text{TiO}_2$  and  $\alpha\text{-Fe}_2\text{O}_3$  for solar water splitting—superior role of 1D nanoarchitectures and of combined heterostructures, *Chem. Soc. Rev.*, 46, 3716-3769.
2. Hejazi, S., Mohajernia, S., Osuagwu, B., Zoppellaro, G., Andryskova, P., Tomanec, O., Kment, S., Zboril, R., Schmuki, P. (2020) On the Controlled Loading of Single Platinum Atoms as a Co-Catalyst on  $\text{TiO}_2$  Anatase for Optimized Photocatalytic  $\text{H}_2$  Generation, *Adv. Mater.*, 32, 1908505

# The effect of cold plasma and fs-laser generated plasma surface treatment on the wettability of high strength steel and the strength of the overlap bonded joint

Z. Weltsch <sup>1,\*</sup>, M. Berczeli <sup>1</sup>

<sup>1</sup> Department of Innovative Vehicles and Materials, John von Neumann University, Kecskemét, Hungary

## Abstract:

The strength and quality of an adhesion bonding technology can be traced back to the bonding of the binder on the surface of the materials to be bonded. In a similar way to characterising the extent to which a liquid can spread over the surface of a material, this spreading can be characterised by the wetting phenomena and the surface energy of the material under test. If a liquid with a known surface tension is dropped onto a material to be tested, it will take on a geometry as a consequence of the wettability of the surface, which will be completely spread out in the case of good wetting, i.e. in the hydrophilic state, while in the bad state it will take the form of a droplet. From the contact point of the droplet we can determine the wetting contact angle of the liquid. The smaller this angle value, the higher the surface energy of the material under test, which predicts that the strength of the adhesion technology applied to the surface will be higher. In most cases, the materials used in the modern automotive industry have poor wetting properties. However, by using the right preparation and surface treatment process, we can influence the wettability values in a way that is favourable to us.

Such mechanical surface treatment processes include ore dressing and granulation, while chemical processes such as etching and soaking are common. Due to the high quality, aesthetic and environmental regulations in the automotive industry, it is preferable to use processes that do not cause aesthetic deterioration. High energy density surface treatments such as plasma and laser beam treatment can be a suitable solution to optimise the bonding of surfaces. These are the most advanced surface treatment methods available to the automotive industry today, but have been little addressed. A particularly interesting area is the development of bonding of high strength steels by cold plasma and femtosecond laser beam treatment. Both surface treatments involve the application of a plasma beam to the steels to be surface treated. In the case of cold plasma jets, the effect is exerted by the jets exiting the surface treatment machine. In the case of the fs laser beam, the laser power of over

1000 MW causes the steel surface layer to change immediately from the solid state to the plasma state, thus treating the surface and producing a special surface texture. In this research, surface treatment experiments were carried out on DP600 high strength steel and the topographical characteristics of the altered surfaces and the effect of the treatments on wetting were investigated. By bonding the hydrophilic surface materials achieved by the treatments with 2K epoxy, interleaved bonds were created, and the shear strength of the bond was determined by tensile testing. The bond strength of the bonded joints of the surfaces interacting with the plasma flow was increased several-fold.

**Keywords:** cold plasma, surface treatment, high strength steels, wettability, Fowkes, adhesive bonding.

## References:

1. Alimov, V. K. *et al.* (2018) 'Surface morphology of F82H steel exposed to low-energy D plasma at elevated temperatures', *Journal of Nuclear Materials*, 510, pp. 366–372.
2. Braceras, I. *et al.* (2018) 'Plasma nitriding of the inner surface of stainless steel tubes', *Surface and Coatings Technology*, 355(October 2017), pp. 116–122.
3. Garcia-Giron, A. *et al.* (2018) 'Combined surface hardening and laser patterning approach for functionalising stainless steel surfaces', *Applied Surface Science*, 439, pp. 516–524.
4. Lis, K. A. *et al.* (2018) 'Inactivation of multidrug-resistant pathogens and *Yersinia enterocolitica* with cold atmospheric-pressure plasma on stainless-steel surfaces', *International Journal of Antimicrobial Agents*, 52(6), pp. 811–818.
5. Williams, D. F. *et al.* (2017) 'Surface analysis of 316 stainless steel treated with cold atmospheric plasma', *Applied Surface Science*, 403, pp. 240–247.

# Influence of cold plasma surface treatment of HIPS and PA6 based nanocomposite polymers on wetting properties and 2K epoxy and 2K hybrid adhesive joints

M. Berczeli<sup>1, \*</sup>, Z. Weltsch<sup>1</sup>

<sup>1</sup> Department of Innovative Vehicles and Materials, John von Neumann University, Kecskemét, Hungary

## Abstract

Today's innovative surface technology processes offer a wide range of surface preparation options, allowing engineers to make major changes to the properties of products. Surface modification can also improve the quality and thus the strength of the bond created by technologies such as bonding, painting, coating, sealing and other processes based on adhesive bonding.

Rapidly evolving materials research processes often result in materials with highly advantageous properties but sub-optimal interfacial properties, thus limiting their adaptability to different technologies. By activating the molecular groups on the surface, wetting properties can be improved, while by changing the microtopography of the surface, roughness and texture properties can be created. An example of a fast, well-automated, clean and flexibly controllable form of surface modification suitable for industrial conditions is plasma surface modification.

Plasma surface modification is a popular topic in polymer technology and is widely used in the surface activation of polymer products. Although it leaves the topography almost intact, leaving its potential for modification untapped, the intensity of the treatment can be optimised by the speed of the plasma jet nozzle feed and its distance from the surface. This avoids the degradation of the HIPS (High Impact Polystyrene) and polyamide matrix material used. In addition to HIPS and PA-6, the effect of the appearance and increasing amount of carbon nanotubes, widely used and studied in industry and academia, was also investigated.

The changes in interfacial phenomena can be investigated using the room temperature quiescent drop method wetting procedure. By measuring the wetting boundary angles during the process using ethylene glycol and distilled water, the results can be used to calculate the surface free energy of the modified surface in mN/m according to Fowkes' interfacial energy theory. To demonstrate the improvement in adhesion of specimens with good wetting surfaces, a series of bonding experiments should be carried out to demonstrate

the changes in strength as a result of surface treatment. In addition, the technical benefits of modifying the topography can be characterised by investigating the block surfaces.

The aim of my research is to improve the bonding technology of HIPS and polyamide matrix nanocomposites, looking for a surface treatment solution that does not limit the bonding technology to the use of difficult to bond materials with low wetting properties.

**Keywords:** cold plasma, surface treatment, high strength steels, wettability, Fowkes, adhesive bonding.

## References:

1. Fanelli, F. and Fracassi, F. (2017) 'Atmospheric pressure non-equilibrium plasma jet technology: general features, specificities and applications in surface processing of materials', *Surface and Coatings Technology*, 322, pp. 174–201. doi: 10.1016/j.surfcoat.2017.05.027.
2. Kehrner, M. *et al.* (2020) 'Cold atmospheric pressure plasma treatment for adhesion improvement on polypropylene surfaces', *Surface and Coatings Technology*, 403(July), p. 126389. doi: 10.1016/j.surfcoat.2020.126389.
3. Kostov, K. G. *et al.* (2014a) 'Surface modification of polymeric materials by cold atmospheric plasma jet', *Applied Surface Science*, 314, pp. 367–375. doi: 10.1016/j.apsusc.2014.07.009.
4. Kostov, K. G. *et al.* (2014b) 'Surface modification of polymeric materials by cold atmospheric plasma jet', *Applied Surface Science*, 314, pp. 367–375. doi: 10.1016/J.APSUSC.2014.07.009.

# **Plasma Tech Session I: Plasma fundamentals / Modelling / Atomic and Molecular Processes**

# Controlling Atmospheric Pressure Plasma Interactions with Solids and Liquids

Kseniia Konina, Mackenzie Meyer, Sanjana Kerketta, Astrid Raisanen, Jordyn Polito  
and Mark J. Kushner

University of Michigan, Ann Arbor, MI 48109-2122 USA, mjkush@umich.edu

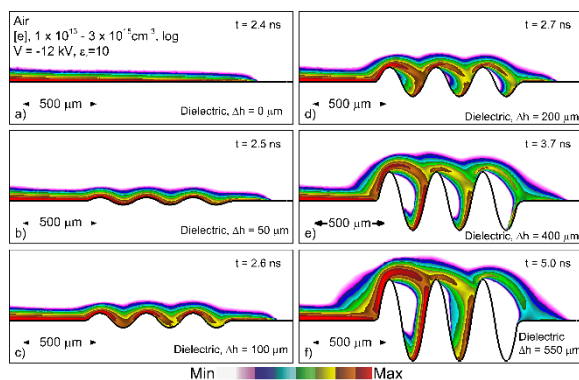
## Abstract:

The use of atmospheric pressure plasmas (APPs) for chemical conversion, plasma catalysis and biomedical applications emphasizes the need for controlling the plasma produced reactant fluxes incident onto surfaces. Unlike low-pressure plasma materials processing, the short mean-free-paths at atmospheric pressure provide a synergistic feedback that can affect the properties of the plasma. Liquid surfaces provide a feedback through evaporation, and changes electrical properties.

In this presentation, results will be discussed from computational investigations of APPs interacting with solid and liquid surfaces. These investigations were conducted with 2-dimensional and global (plug flow) modeling platforms which include plasma and gas dynamics, radiation transport, and surface and liquid chemistry.

Plasma interactions with several classes of surfaces will be discussed: a) electrolytic solutions as used in materials synthesis, b) polymers, c) biomedical solutions and d) complex (nonplanar) surfaces. Each class of surface presents particular challenges. For example, when treating complex (non-planar) dielectric surfaces, the charging and polarization of the surface feeds back to the plasma, making uniform exposure of the surface difficult. (See Figure 1.) The plasma induced properties of biomedical solutions are sensitive functions of the entrainment of air into the plasma plume, that in turn effects pH and the biocidal properties of the solution. The conductivity of electrolytic solutions affects plasma properties through limiting current similar to

dielectric barrier discharges. Limiting current then determines the rate of electron solvation, an important parameter in the reduction of ions for materials synthesis.



**Figure 1:** A negative, atmospheric pressure (humid air) surface ionization wave (SIW) propagating across a wavy dielectric surface. The SIW detaches from the surface by launching new bulk ionizations waves due to electric fields produced by surface charging and polarization of the surface.

**Keywords:** atmospheric pressure plasma, plasma-surface interactions, plasma-liquid interactions, radiation transport, modeling, simulation

## Acknowledgement:

This work was supported by the US Department of Energy Office of Fusion Energy Sciences, National Science Foundation and Department of Defense MURI (Multidisciplinary University Research Initiative) Program.



# Plasma spraying related modelling and experimental studies

M. Baeva\*, T. Zhu, H. Testrich, R. Methling, R. Foest

Leibniz Institute for Plasma Science and Technology, Greifswald, Germany

## Abstract:

Plasma spray torches are widely used in industrial applications concerning the deposition of protective and functional coatings. The quality and the properties of the produced coatings result from the processes on the entire chain: the operation conditions of the plasma torch, which are related to the characteristics of the electric arc and the generated plasma jet; the feeding of the powder material and its behavior in the plasma jet; the deposition of the molten particles on the target.

This work presents an overview of the modelling and experimental studies carried out on a plasma spray torch and on the processes in the plume of the plasma jet, where the powder material is injected (Figure 1).

The electric and thermal characteristics of the direct current plasma torch F4MB-XL (Oerlikon Metco) are studied for a steady operating mode by means of a magneto-hydrodynamic (MHD) model [1] that couples a submodel of a refractory cathode and its non-equilibrium boundary layer to a submodel of the plasma in local thermodynamic equilibrium in a self-consistent manner. It was found out that an appropriate description of the flow accounting for Mach numbers beyond 0.3 is essential for the agreement of the model predictions with experimental findings.

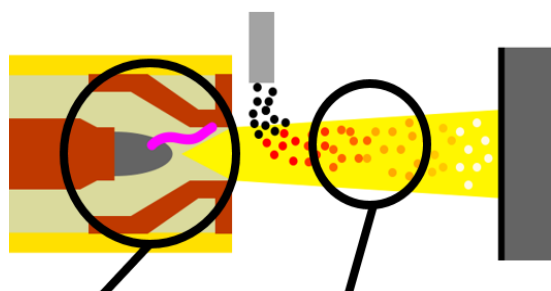
The simulation results are used to analyze the erosion of the refractory cathode made of doped tungsten [2]. A model of the ionization layer of the cathode and the evaporation of tungsten atoms is considered in order to study the transport of the evaporated atoms and to estimate the loss of cathode material. The model's prediction was found to be in a fair agreement with measured values.

The behavior of the powder material injected into the plume of the plasma jet is studied by means of a model for particle tracing. This model accounts for heating and melting of the particles and allows one to obtain the particle trajectories, velocities, and temperatures. Statistical distributions of the particle size, angle and velocity of injection based on the real experimental conditions are taken into account.

The modelling and experimental studies are carried out in the framework of a project funded by

the European Union and the Federal State of Germany Mecklenburg—Western Pomerania (Project number TBI-V-1-321-VBW-112).

**Keywords:** plasma spray torch, magneto-hydrodynamic simulation, transport of evaporated material, cathode erosion, particle tracing, electric and thermal measurements.



**Figure 1:** This figure explains the main parts of the study that will be presented: the MHD simulation of the plasma torch and the determination of the arc parameters; the modelling of cathode erosion; the tracing of powder particles fed in the plasma jet for spraying, experimental findings concerning the electric and thermal torch characteristics, properties of the powder particles.

## References:

1. Baeva, M., Zhu, T., Kewitz, T., Testrich, H., Foest, R., Journal of Thermal Spray Technology (2021), DOI 10.1007/s11666-021-01261-4.
2. Baeva, M., Benilov, M. S., Zhu, T., Testrich, H., Foest, R.: Modelling and experimental evidence of the cathode erosion in a plasma spray torch, Journal of Physics D: Applied Physics (to be submitted).

# Non-Thermal Plasma Sources Based on Cometary and Point-to-Ring Discharges

Eliška Lokajová<sup>1</sup>, Josef Khun<sup>1</sup>, Anna Machková<sup>1</sup>, Petra Kašparová<sup>1</sup>, Myron Klenivskyi<sup>1,\*</sup>, Eva Vaňková<sup>1</sup>, Pavel Galář<sup>2</sup>, Jaroslav Julák<sup>1,3</sup> and Vladimír Scholtz<sup>1</sup>

<sup>1</sup>Department of Physics and Measurements, University of Chemistry and Technology, 16628 Prague, Czech Republic

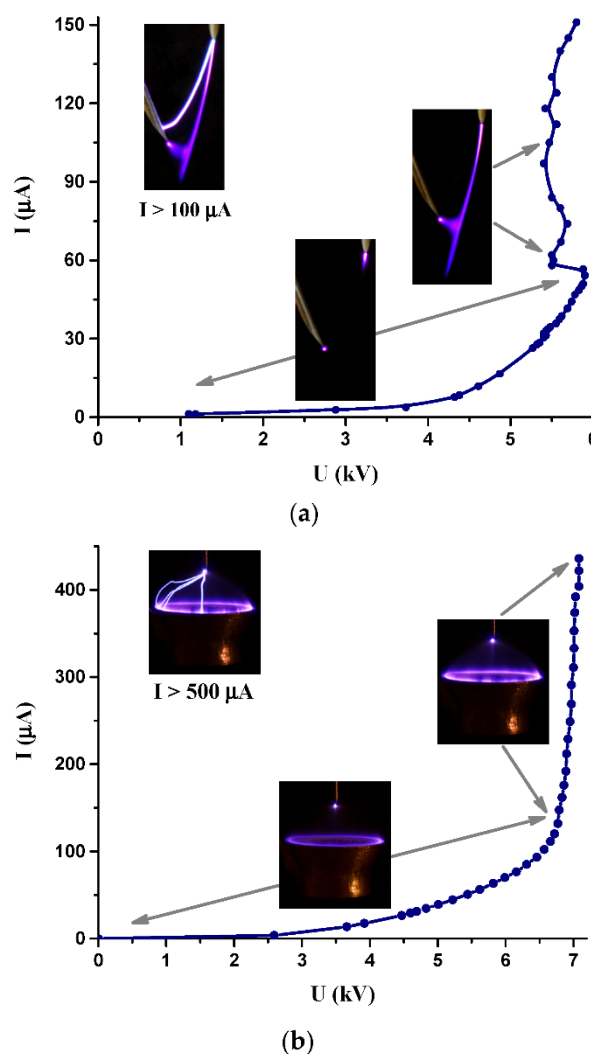
<sup>2</sup>Department of Thin Films and Nanostructures, Institute of Physics, Czech Academy of Sciences, 16200 Prague, Czech Republic

<sup>3</sup>Institute of Immunology and Microbiology, First Faculty of Medicine, Charles University and General University Hospital, 12800 Prague, Czech Republic

## Abstract:

A Non-thermal plasma (NTP) is a promising tool against the development of bacterial, viral, and fungal diseases. The recently revealed development of microbial resistance to traditional drugs has increased interest in the use of NTPs. We have studied and compared the physical and microbicidal properties of two types of NTP sources based on a cometary discharge in the point-to-point electrode configuration and a corona discharge in the point-to-ring electrode configuration (Figure 1). The electrical and emission properties of both discharges are reported. The microbicidal effect of NTP sources was tested on three strains of the bacterium *Staphylococcus aureus* (including the methicillin-resistant strain), the bacterium *Pseudomonas aeruginosa*, the yeast *Candida albicans*, and the micromycete *Trichophyton interdigitale*. In general, the cometary discharge is a less stable source of NTP and mostly forms smaller but more rapidly emerging inhibition zones on agar plates. Due to the point-to-ring electrode configuration, the second type of discharge has higher stability and provides larger affected but often not completely inhibited zones. However, after 60 min of exposure, the NTP sources based on the cometary and point-to-ring discharges showed a similar microbicidal effect for bacteria and an individual effect for microscopic fungi.

**Keywords:** *Candida albicans*; corona discharge; *Pseudomonas aeruginosa*; microbicidal effect; *Staphylococcus aureus*; *Trichophyton interdigitale*



**Figure 1:** Figure illustrating the volt–ampere characteristics and relevant images of the cometary discharge point-to-point (a) and point-to-ring discharge (b)

## References:

Khun, J.; Machková, A.; Kašparová, P.; Klenivskyi, M.; Vaňková, E.; Galář, P.; Julák, J.; Scholtz, V. Non-Thermal Plasma Sources

Based on Cometary and Point-to-Ring Discharges. *Molecules* **2022**, *27*, 238.

<https://doi.org/10.3390/molecules27010238>

# A modelling investigation of the mechanisms underlying the O/O<sub>2</sub> removal in the afterglow of a CO<sub>2</sub> plasma with a carbon bed

Omar Biondo<sup>1,2</sup>, Fanny Girard-Sahun<sup>1</sup>, Georgi Trenchev<sup>1</sup>, Gerard van Rooij<sup>2,3</sup> and Annemie Bogaerts<sup>1</sup>

<sup>1</sup>Research Group PLASMANT, Department of Chemistry, University of Antwerp, Universiteitsplein 1, Wilrijk B-2610, Belgium

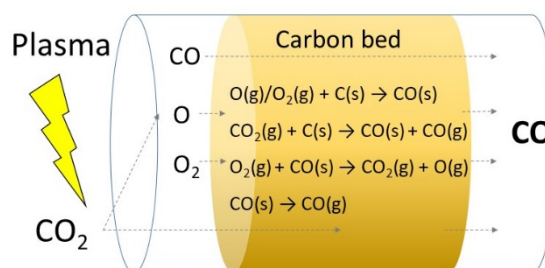
<sup>2</sup>DIFFER, 5612AJ Eindhoven, The Netherlands

<sup>3</sup>Faculty of Science and Engineering, Maastricht University, 6229 GS Maastricht, The Netherlands

## Abstract:

In recent years, there is a growing interest for the potential of plasma technologies to split CO<sub>2</sub> into CO and O and convert it into value-added products. Oxygen atoms in the effluent may trigger back reactions, reducing the overall yield and efficiency, as well as are incompatible with desired follow-up chemistry such as Fischer-Tropsch<sup>1,2</sup>. In this regard, a promising route is to use solid carbon (e.g. biochars) after the plasma reactor, which can be oxidized by O and/or O<sub>2</sub> to produce CO, further increasing the CO yield, and removing the O<sub>2</sub> from the product mixture. Although it has been experimentally proven that carbonaceous materials are effective options, more insight is needed to demonstrate their full potential and identify the relevant mechanisms underlying the O<sub>2</sub> removal. In this respect, modelling is a very useful tool to predict the chemistry, especially when this is inaccessible experimentally. Zero-dimensional (0D) chemical kinetic models are the method of choice to study the contributions of different chemical reactions occurring in plasma ignited in complex gas mixtures<sup>3</sup>. Therefore, we validate a 0D kinetic model with gasification experiments, in order to get insights into the reaction pathways for carbon oxidation in the presence of O<sub>2</sub>, CO<sub>2</sub> and CO and the deactivation of the carbon bed, with consequent decrease in selectivity towards the latter.

**Keywords:** CO<sub>2</sub> conversion, CO<sub>2</sub> utilization, carbon gasification, plasma, reverse Boudouard reaction, oxygen removal, charcoal, modelling, detailed chemistry.



**Figure 1:** Schematic of the mechanism of carbon gasification in the post CO<sub>2</sub> plasma region, leading to enrichment of CO in the product mixture.

## References:

1. Aerts, R., Snoeckx, R., Bogaerts, A. (2014) In-Situ Chemical Trapping of Oxygen in the Splitting of Carbon Dioxide by Plasma: In-Situ Chemical Trapping of O<sub>2</sub> in CO<sub>2</sub> Splitting by DBD, *Plasma Process. Polym.*, 11, 985–992.
2. van Rooij, G.J., Akse, H.N., Bongers, W.A., van de Sanden, M.C.M. (2018) Plasma for electrification of chemical industry: a case study on CO<sub>2</sub> reduction, *Plasma Phys. Control. Fusion*. 60, 014019.
3. Alves, L.L., Bogaerts, A., Guerra, V., Turner, M.M. (2018) Foundations of modelling of nonequilibrium low-temperature plasmas. *Plasma Sources Science and Technology*. 27 (2), 023002.

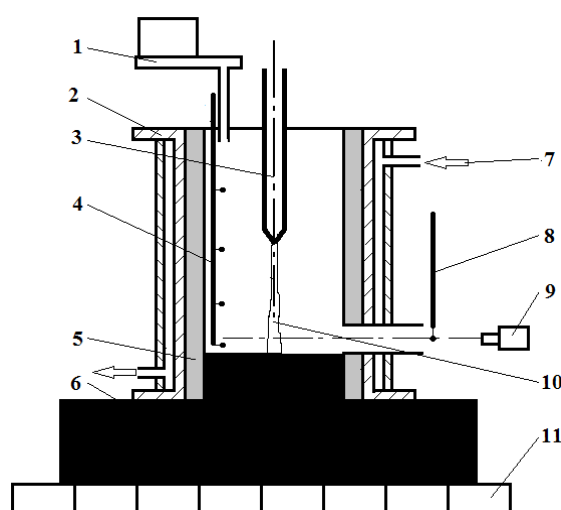
## Behaviour of Electric Arc in Plasma-Chemical Reactor During Hazardous Waste Processing

V. Grigaitienė, D. Gimžauskaitė, V. Valinčius, R. Kėželis, R. Uscila, Ž. Kavaliauskas

Lithuanian Energy Institute, Plasma Processing Laboratory, Lithuania

### Abstract:

This paper presents the electrical characteristics and optical images of DC electric arc behaviour in plasma chemical reactor (PChR) during various hazardous waste processing (Figure 1). A novelty of this research is the employment of atmospheric pressure DC plasma torch with a free burning electric arc for waste treatment in a volume reactor [1].



**Figure 1:** The schematic representation of plasma-chemical reactor. 1 – raw material input, 2 – cooled block, 3 – plasma torch, 4 – probe for gas temperature measurements, 5 – inner high-temperature insulation layer, 6 – graphite anode, 7 – cooling water in, 8 – probes for outlet gas temperature, content and spectra measurements, 9 – high-speed camera, 10 – arc column, 11 – insulating base.

During the waste treatment, the liquid phase of recyclable materials and the gas phase with very fine solids particles exist in the reactor. The amount and size of melted domains and particles mainly depend on waste nature and arc column parameters. Plasma forming gas with a high concentration of fine solids particles in the surrounding of the arc causes the destabilisation

and fluctuations inside the reactor. This affects the current, voltage and shape of the electric arc column, which in turn determines the unstable PChR operating in general. The electric arc can diffuse in different areas and places of the reactor chamber. In turn, these effects change the shape and burning area of the arc spot, which greatly influences the energy transfer to recycling material. Since the amount of experimental data on arc characteristics and parameters during plasma processes is limited, this paper presents the results of an experimental investigation on the influence of waste nature and composition on the electric arc characteristics in PChR at atmospheric pressure. The conditions for stable arc operating in plasma chemical reactor during various waste treatment at atmospheric pressure, their spatial, electrical and optical characteristics are established. It has been shown that the stable arc burning process depends also on arc column length. Results, obtained during the processing of various mixed wastes, show the capability to recycle them into nonhazardous molten and gaseous states using a plasma chemical reactor.

**Keywords:** plasma, plasma chemical reactor, electric arc, hazardous waste

### References:

1. Valinčius, V., Kėželis, R., Gimžauskaitė, D., Grigaitienė, V., Valatkevičius, P. The investigation of an electric arc in a plasma chemical reactor for hazardous waste treatment. *Journal of Chemical Technology and Biotechnology* Vol 95, (2020) 450-456.

### Acknowledgement

This research (project no. 01.2.2-LMT-K-718-01-0069) is funded by the European Regional Development Fund according to the supported activity 'Research Projects Implemented by World-class Researcher Groups' under measure no. 01.2.2-LMT-K-718.

# Global plasma modelling of PECVD growth of SiO<sub>2</sub> films for optical applications

K. Tomankova<sup>1,2</sup>, N. Rivolta<sup>3</sup>, A. Obrusnik<sup>1</sup>

<sup>1</sup> PlasmaSolve company, Brno, Czech Republic

<sup>2</sup> Masaryk University, Brno, Czech Republic

<sup>3</sup> AGC-Technovation Center, Charleroi, Belgium

## Abstract:

This contribution presents a simulation study of plasma-enhanced chemical vapour deposition (PECVD) of SiO<sub>2</sub> from HMDSO (hexamethyldisiloxane) in large-scale industrial coaters for glass applications. Usage of such complex precursors (e.g. in optical coatings) leads to a plethora of gas phase reactions dominating the process. As the size of the precursor molecule gets more complex, the number of possible reaction pathways grows exponentially. Especially concerning organic molecules, these could theoretically undergo thousands of different reactions. It is not straightforward to assess which of these processes are the most influential and which can be of secondary relevance. The importance of individual processes is often application-specific or even coater-specific.

In this work we investigate the characteristics of a plasma ignited in oxygen and HMDSO admixtures used for depositions of SiO<sub>2</sub>-like films. We consider a reactor consisting of 4 hollow cathode plasma sources (HC) connected to a deposition chamber. O<sub>2</sub> plasma is ignited in the HC and the discharge leaks into the deposition region, where HMDSO is added to the process. Each HC is powered by 4-6kW, the O<sub>2</sub> flows into the HC with 300-600sccm which results in pressure in the order of 10<sup>2</sup>Pa. In the beginning, we developed a 0D global plasma model (GPM), that characterizes the plasma discharge in the HC. The time-averaged GPM results of densities and temperature were compared to Langmuire probe measurements of the same O<sub>2</sub> discharge in a similar reactor in Germany. Our GPM results exhibited very similar trends to the measurements. We also utilize the GPM to study the scaling parameters of hollow cathode plasma systems in oxygen.

Along with the O<sub>2</sub> GPM we are currently developing a spatially-resolved 2D simulation of the same plasma to get another benchmark for the results and gain knowledge on the spatial distributions of the plasma.

Finally, we developed a HMDSO GPM coupled to the O<sub>2</sub> GPM, meaning the previous results present the input of HMDSO GPM. This model describes the complex O<sub>2</sub>/HMDSO chemistry with 1209 reactions and processes involving numerous carbon, hydrocarbon and oxygen species obtained from (Kemaneci et al., 2019). With this model we aim to find optimal plasma parameters to promote the formation of SiO<sub>2</sub>-like molecules. First GPM results and coating analysis from our geometry and a similar one in Germany suggest, that for an effective growth of SiO<sub>2</sub>-like films it is efficient to dissociate HMDSO molecule with atomic oxygen, rather than electrons, to trigger the formation of Si-O-Si bonds and minimise creation of hydrocarbon radicals. We also perform sensitivity analysis on the GPM in order to exclude the most influential reactions out of the large and general kinetic system of 1209 reactions. These will then be used in spatially-resolved 2D plasma models.

## Acknowledgement

This work is carried out within the Multi-Scale Simulation Toolbox (MIST) project funded within M-era.net 2 joint call (Reference Number: project8261).

This work is made in collaboration with AGC-Technovation Center.

**Keywords:** PECVD, hollow cathode, HMDSO, SiO<sub>2</sub>, optical coatings, reactive plasma modelling, global plasma model.

## References:

Kemaneci, E., Mitschker, F., Benedikt, J., Eremin, D., Awakowicz, P., & Brinkmann, R. P. (2019). A numerical analysis of a microwave induced coaxial surface wave discharge fed with a mixture of oxygen and hexamethyldisiloxane for the purpose of deposition. *Plasma Sources Science and Technology*, 28(11). <https://doi.org/10.1088/1361-6595/ab3f8a>



## Characterization of non-thermal coronal plasma discharges for different high voltage electrode configurations in the atmosphere using air.

S.O. Babalola<sup>1</sup>, V. Tshigo<sup>1</sup>, M.O. Daramola<sup>1</sup>, S.A. Iwarere<sup>1\*</sup>

<sup>1</sup>Department of Chemical Engineering, Faculty of Engineering, Built Environment and Information Technology, University of Pretoria, Hatfield 0028, Pretoria, South Africa.

\*Corresponding author: [samuel.iwarere@up.ac.za](mailto:samuel.iwarere@up.ac.za)

### Abstract:

The generation of fast reactive species has been a key factor that renders non-thermal plasma technology highly effective, cost-effective, and environmentally friendly in water treatment processes. Morphological imaging of a high voltage AC coronal filamentary and diffuse discharge is characterized for two distinct high voltage electrode configurations for the same polarity. The dependency of these phenomena on reactor inputs such as water flow rate, water temperature, input voltage, and frequency are investigated and their effects on the reactor's capability to generate high reactive species are highlighted. The use of sharp concave high electrodes for the generation of plasma has been emphasized in literature as the most effective shape for generation of plasma. Therefore, this work aims to further expound on this narrative and provide substantial evidence to support further investigation of simple high electrode design such as a shaft with a finished surface by analyzing the plasma generation quality in these two electrodes for the same reactor design. This work further highlights the distribution of the diffuse discharge on the reactor surface for varying water flow rates and marries this property to filamentary discharge propagation within the reactor in the case of smooth shaft electrode.

**Keywords:** diffuse discharge, filamentary discharge, morphological imaging, coronal discharge, Pulsed frequency.

# Investigations of the sheath in a dual-frequency capacitively coupled rf discharge by optically trapped microparticles

J. Schleitzer<sup>1</sup>, V. Schneider<sup>1</sup> and H. Kersten<sup>1</sup>

<sup>1</sup> Institute of Experimental and Applied Physics, Christian-Albrechts-University, Kiel, Germany

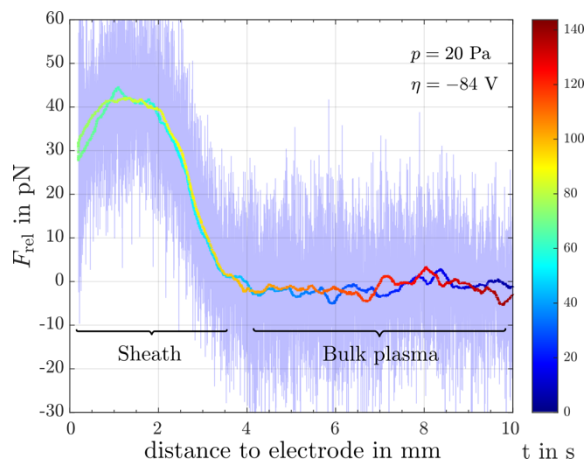
## Abstract:

Many different diagnostics can be used to measure the spatial and temporal evolution of plasma parameters. During the last decade, the idea to use externally injected, small microparticles as non-invasive probes, which are influenced by various forces and energy fluxes in plasmas, was implemented. Based on their behavior in the surrounding plasma, information about local electric fields [1], energy fluxes towards the particle [2] and momentum transfer by ions to the particles [3] can be obtained. Especially the manipulation of microparticles by an optical tweezer is of great interest, since it allows the microprobe to be moved into areas of the plasma, which are usually not accessible by common diagnostic methods, i.e. the plasma sheath.

In this study, optically trapped microparticles in an optical tweezer are used to investigate the sheath of a dual-frequency CCRF discharge. This discharge is known in particular for its ability to control the ion flux and the ion energy separately and independently based on the utilization of electrical asymmetries in the plasma [4,5].

The measured quantity, which is of special importance when using optical tweezers, is the external force acting on the microprobe. It is obtained by measuring the displacement of the particle in the optical trap while the confined microprobe will be moved through the plasma and the sheath and, thus, relatively to the discharge [6]. On the basis of these force profiles (Figure 1), the strength of the electric field force in the sheath as a function of pressure and the dependence on the distance of the probe to the rf electrode as well as the extent of the sheath are determined, both in a single- and a dual-frequency discharge [7].

**Keywords:** phase variation, radiofrequency discharges, signal detectors, optical tweezers, microprobe, physical quantities, optical trapping, plasma sheaths, plasma diagnostics, force measurement, electrical asymmetry effect.



**Figure 1:** Figure showing the measured relative force profile obtained by the use of an optically trapped microparticle for a certain pressure  $p$  and a specific dc self-bias  $\eta$  in a dual-frequency discharge. The colored curve represents an averaging over the raw data shown and the color gradient illustrates the time required for the measurement.

## References:

1. E. B. Tomme et al., Phys. Rev. Lett. 85(2000), 2518-2521.
2. H. Maurer, R. Basner, and H. Kersten, Rev. Sci. Instrum. 79(2008), 093508.
3. V. Schneider et al., Rev. Sci. Instrum. 81(2010), 013503.
4. Z. Donkó, J. Schulze, B. G. Heil, and U. Czarnetzki, J. Appl. Phys. 42(2008), 025205.
5. J. Schulze, E. Schüngel, and U. Czarnetzki, J. Phys. D: Appl. Phys. 42(2009), 092005.
6. V. Schneider and H. Kersten, Rev. Sci. Instrum. 89(2018), 103505.
7. J. Schleitzer et al., Phys. Plasmas 28(2021), 083506.

## **SICT 2022 / Tribology 2022:**

### **ReSISTant EU Project Workshop: Drag Reducing Surfaces (Riblets)**

**- Simulations, high resistant  
nano-coatings development,  
applications, and new  
manufacturing processes for  
future worldwide applications**

# Overview Project ReSiSTant

A. Flanschger<sup>1</sup>

<sup>1</sup> Research and Development, bionic surface technologies GmbH, Graz, Austria

## Abstract:

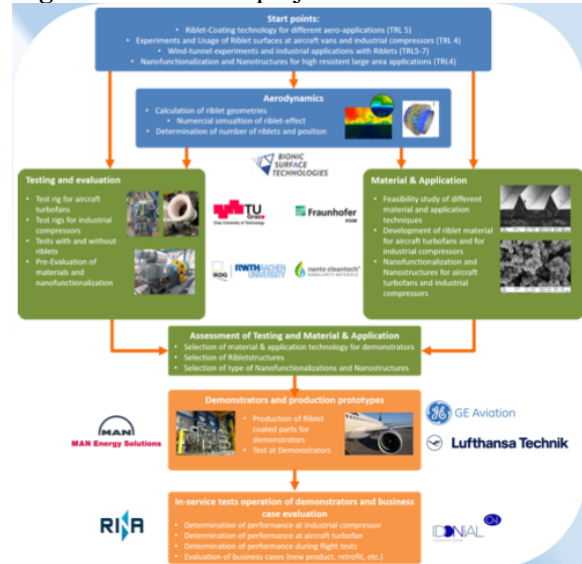
ReSiSTant project targets the optimization of two industrial pilot lines by using micro- and nanostructured surfaces for drag reduction. The objectives are to implement new developed surfaces into:

### 1) Aircraft Turbofan Engines

### 2) Industrial Compressors.

Positive effects by usage of such surface could give benefits in terms of efficiency, CO<sub>2</sub> reduction and noise emission and further on a positive economic and ecological impact.

**Figure 1:** Overview project ReSiSTant



**Keywords:** drag reduction, efficiency increase, microstructured surfaces, turbofan jet engine, industrial compressors

## Industrial Compressors: Riblet Simulation Results

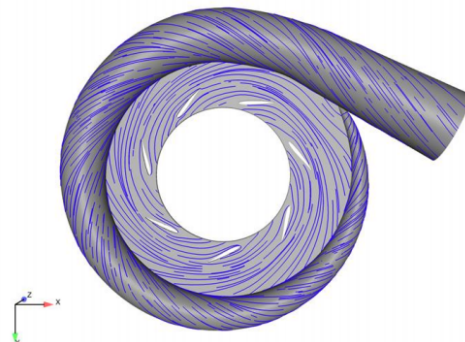
Mr. Lucas Garcia de Albeniz Martinez;

Bionic Surfaces Technologies GmbH, Graz, Austria

### Abstract:

Microstructured surfaces (so-called Riblet surfaces or 'shark-skin') reduce the frictional resistance of flat surfaces by up to 8% when the trapezoidal shape is used. In the frame of ReSiSTant Project, their effect on a centrifugal gas compressor is studied. Theoretical, numerical and experimental experiences give a first estimation of the impact of Riblets. In this case, the Riblet structures reduce the aerodynamic shear stress losses. The areas of higher interest are the diffuser and the volute. In those areas, the viscous losses are large and the use of Riblets could bring a big benefit. The optimal size, position and effect on performance are going to be analyzed. The ultimate goal of the project is to study how big is the impact of the ideal Riblets applied on a centrifugal gas compressor.

**Keywords:** CFD, Shark skin effect, Industrial Compressor.



**Figure 1:** Figure illustrating the optimal riblet direction applied on different parts of a centrifugal compressor: diffuser and volute

## Industrial Compressors: Applications and Test results

M. Meyer  
MAN Energy Solutions Switzerland AG

**Abstract:**

In the course of the project “ReSISant”, a new coating was developed by NantoClean Tech. With this coating very smooth surfaces can be achieved which are also resistant against erosion. In order to test this, the coating was applied on the diffuser of a radial industrial compressor. Two sets of measurements were carried out: One before and one after applying the coating. The efficiency increase was substantial, but had to be evaluated, since this measured increase originates partly through the cleaned surface and partly through the coating.



## Riblet application technologies Coatings for harsh conditions

Andreas BRINKMANN<sup>1</sup>, Yvonne KOWALIK<sup>1</sup>

<sup>1</sup> Fraunhofer Institute for Manufacturing Technology and Advanced Materials IFAM, Wiener Straße  
12 28359 Bremen, Germany, [andreas.brinkmann@ifam.fraunhofer.de](mailto:andreas.brinkmann@ifam.fraunhofer.de)

### **Abstract**

Microstructured surfaces are sometimes used in highly stressed environments. In the EU project ReSISTant, environments are tested, which have enormous temperatures. This restricts the selection of possible coating systems enormously. Different materials will be presented, which can be used for different loads and applications.

## Riblet Application Technologies : Novel Free Form Riblet Processing Technology by Laser

Yuichi Shibasaki, Nikon Corporation

### **Abstract:**

Novel free form riblet processing technology using ultra short pulse laser is introduced. This technology enables the following features a) aerodynamically ideal freeform riblet, b) direct engraving in almost all materials, c) processing on 3-dimensional curved surfaces, d) good precision.

# Riblet Application Technologies: Direct Contactless Microfabrication

H. Bilinsky<sup>1</sup>

<sup>1</sup> MicroTau, Sydney, Australia

## Abstract:

MicroTau's Direct Contactless Microfabrication (DCM) technology grows riblets with light by drawing on photolithographic techniques developed for computer chip fabrication. Using tailored microstructured light patterns, riblets are grown on a substrate, with the riblet size and profile determined by the exposure conditions. Advancements in this technology, including improvements to riblet geometry resulting in MicroTau manufacturing high aspect ratio 'blade' type riblet designs not possible to manufacture with alternative technologies at the scale required for aviation and maritime applications. Being a contactless optical printing process DCM can rapidly iterate 3D riblet designs for nominal cost in contrast to typically expensive tooling costs associated with NIL and other contact manufacturing processes. Recent developments include manufacturing scale up and flight testing of riblet film product manufactured with DCM.

**Keywords:** drag reduction, shark skin, riblets, riblet, microstructures, fabrication, manufacturing, microsurfaces, efficiency, fuel efficiency, photolithography, DCM, aircraft.



**Figure 1:** Optical micrograph of the cross-section of the improved riblet structures from PDMS moulded copy of the riblets. These are riblets with spacing 78  $\mu\text{m}$ , height 41  $\mu\text{m}$ , width 7  $\mu\text{m}$ , peak radius 0.8  $\mu\text{m}$ .

## Riblet Material Development Antifouling Riblet coatings

Andreas BRINKMANN<sup>1</sup>

<sup>1</sup> Fraunhofer Institute for Manufacturing Technology and Advanced Materials IFAM, Wiener Straße  
12 28359 Bremen, Germany, [andreas.brinkmann@ifam.fraunhofer.de](mailto:andreas.brinkmann@ifam.fraunhofer.de)

### **Abstract**

Biological growth on surfaces causes problems in many technical applications and vehicles. Microstructured surfaces are especially affected because the positive properties of the structuring are lost through the growth of a biofilm. In the presentation examples are given of how materials can be equipped to prevent biological growth and protect the structured surfaces.

# **Plasma Tech Session II:**

## **Plasma Processing / materials interactions / coatings**

# Selectively changing key surface properties via atmospheric gliding arc plasma deposition

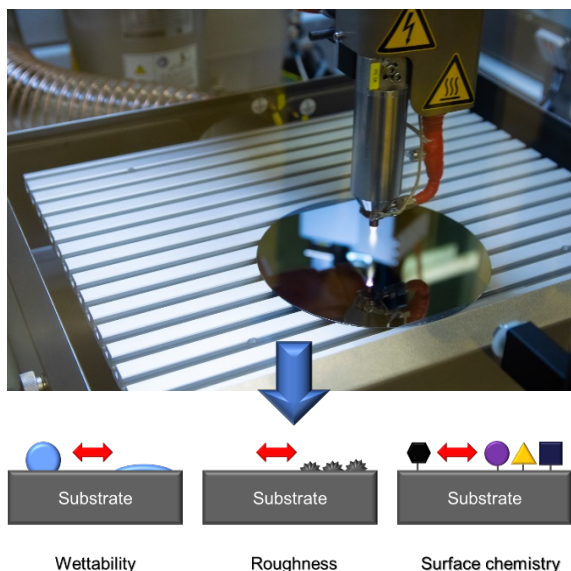
Thomas Danny Michl <sup>1\*</sup>, Alok Goel <sup>1</sup>, Sonja Neuhaus <sup>1</sup>

<sup>1</sup> Fachhochschule Nordwestschweiz FHNW, Hochschule für Technik, Institut für Nanotechnische Kunststoffanwendungen, Klosterzelgstrasse 2, 5210 Windisch, Switzerland

## Abstract:

Controlling the three key surface properties, wettability, roughness and surface chemistry, is key to many industrial applications. Such industrial applications can range from glueing, inking, corrosion control to selective attachment of active molecules on the surfaces. Moreover, it is crucial to control these three surface properties without changing the material's bulk properties. Recently, atmospheric plasma processes have leapt forward and thus gained widespread attention. Like the low-pressure plasma processes, atmospheric plasmas are fast, solvent-free and environmentally friendly. However, unlike their low-pressure counterparts, atmospheric plasmas can be readily implemented into existing production lines and easily scaled up. Specifically, atmospheric gliding arc plasma (AGAP) combines homogeneity and high power densities in comparison to other atmospheric plasma methods. We hereby demonstrate how a commercially available system (Plasmatreat<sup>®</sup>) can be used to rapidly deposit novel thin film coatings. These thin-film coatings selectively change surface wettability, roughness and chemistry. Furthermore, this deposition requires only electricity, benign & readily available gases and small amounts of monomer precursor. By avoiding the use of noble gases or toxic precursors, contrary to other atmospheric plasma methods, AGAP is a viable and safe option to run on an industrial scale.

**Keywords:** Surface engineering, surface modification, thin film coatings, atmospheric plasma, gliding arc plasma, wettability, roughness, surface chemistry



**Figure 1:** Atmospheric gliding arc plasma deposition can selectively change the three main properties of surfaces; wettability, roughness and surface chemistry

## References:

1. [https://www.plasmatreat.com/plasma-treatment/plasma-pretreatment/plasma-coating\\_nano\\_coating.html](https://www.plasmatreat.com/plasma-treatment/plasma-pretreatment/plasma-coating_nano_coating.html)



# On the formation of carbon nanoparticles in expanding laser-induced plasma

A. Kaczmarek<sup>1,\*</sup>, J. Hoffman<sup>1</sup>

<sup>1</sup> Department of Experimental Mechanics, Institute of Fundamental Technological Research Polish Academy of Sciences, Warsaw, Poland

\*akaczmar@ippt.pan.pl

## Abstract:

Nowadays, there are numerous works devoted to characterizing the properties of carbon nanoparticles obtained by laser ablation in various media, e.g. in liquid [1]. Therefore, it is possible to optimize and control the size of particles, their optical and morphological properties and link them with synthesis parameters, such as the kind of liquid medium or laser fluence. However, the description of primary formation processes of carbon nanoparticles after laser ablation in vacuum, gas or liquid remains incomplete [2].

There have been several attempts to describe the formation of carbon nanoparticles. One of them [3] considered the entire lifecycle of carbon ablation plasma. According to this study, nanoparticles are formed as a result of the ejection of fragments of the target (graphite). Another possibility is ejection of liquid droplets as an effect of so called phase explosion and explosive boiling [4]. Other studies postulate that nanoparticles are formed during plasma-phase expansion [2, 5] and that this process can be divided into two two main phases: (1) nucleation and (2) growth and crystallization.

The aim of this work is to analyze possible nucleation and growth paths of particles in carbon plasma. The consideration of nucleation type is based on the following equation:

$$\Delta G = -\frac{4\pi r^3}{3V} kT \ln S + 4\pi r^2 \gamma,$$

where  $\Delta G$  is Gibbs free energy,  $r$  nucleus size,  $V$  volume occupied by the single atom or molecule in nucleus,  $T$  is temperature,  $S$  supersaturation ratio and  $\gamma$  surface tension [2].

Further modifications to the above equation are made to account for ion nucleation. It has been found that in the case of carbon plasma the purely thermodynamic approach of continuous media is insufficient. It is necessary to include in the considerations various kinds of carbon macromolecules.

Moreover, the discussion of the conditions for the formation of particles solely from the expanding plasma and the avoidance of their ejection from the target is presented.

**Keywords:** nanoparticles, carbon dots, laser ablation, nucleation, ion-induced nucleation.

## References:

1. Małolepszy, A., Błoński, S., Chrzanowska-Giżyńska, J., Wojasiński, M., Płociński, T., Stobiński, L. (2018), Szymański Z., Fluorescent carbon and graphene oxide nanoparticles synthesized by the laser ablation in liquid, *Appl. Phys. A-Mater.*, 124, 228-1-7.
2. Taccogna, F. (2015), Nucleation and growth of nanoparticles in a plasma by laser ablation in a liquid, *J. Plasma Phys.*, 81, 495810509.
3. Harilal, S.S., Hassanein, A., Polek, M. (2011), Late-time particle emission from laser-produced graphite plasma, *J. Appl. Phys.*, 110, 053301.
4. Bulgakova, N.M., Bulgakov, A.V. (2001), Pulsed laser ablation of solids: transition from normal vaporization to phase explosion, *Appl. Phys. A*, 73, 199–208
5. Reznikov, B.I., Alekseev, N.I., Bobashev, S.V., Zhukov, B.G., Ponyaev, S.A., Kurakin, R.O., Rozov, S.I. (2011), Equilibrium composition and the effective adiabatic exponent of a clustering carbon plasma. *Tech. Phys.*, 56, 1106.

# Carbon nanostructure production from ethanol by cold plasma

A. Jurov <sup>1,2,\*</sup>, J. Zavašnik <sup>1</sup>, U. Cvelbar <sup>1</sup>

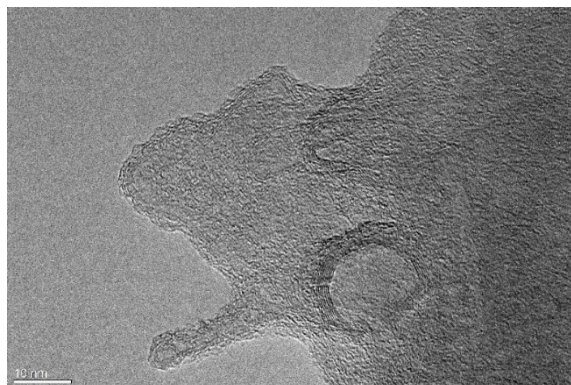
<sup>1</sup>Department F6, Jožef Stefan Institute, Ljubljana, Slovenia

<sup>2</sup>Faculty of Chemical Engineering and Technology, University of Zagreb, Zagreb, Croatia

## Abstract:

The conversion of liquids into some kind of materials has always been highly interesting. Since carbon is an abundant material, it is of special interest for this type of application. Carbon can be organised in various forms of nanostructures like carbon nanotubes (CNTs), graphene, carbon black, nanodiamonds, fullerene, and many more. Some of these carbon allotropes have special properties that make them interesting, and perhaps graphene and carbon nanotubes are the most motivating. Even though there are several synthesis paths of these nanostructures, there is always a need to improve and find alternative options. One of the options that have been recently explored is synthesis from liquids with plasma assistance. Liquids of interest are alcohols since they are carbon-based and are affordable and accessible, especially ethanol. This synthesis pathway suppresses precursors in liquids, including impurities, making processes easier, faster, cheaper, and more environmentally friendly. According to some recently published studies, nanocarbon synthesis in ethanol is mostly produced by in-liquid plasmas called sparks, arcs <sup>1,2</sup>, etc. We decided to use the simpler model setup with an atmospheric pressure plasma jet, where plasma forms in the gas phase over a liquid surface and interacts with liquid. So far, the experiments have been done in ethanol, and obtained results have indicated a fast pathway to convert ethanol into amorphous nanosheets. With increased treatment time, graphene forms on the edges of obtained carbon nanosheets. With this process, the micro and nanostructures were produced in a matter of minutes by the degradation and conversion of ethanol. Additionally, we found that when a graphite sheet is submerged in ethanol, the size of produced structures is reduced, and structured carbon is synthesised. The next step is to tailor conversion pathways with the optimisation of plasma parameters.

**Keywords:** carbon, nanostructures, graphene, ethanol, atmospheric pressure plasma, cold plasma, plasma-liquid interaction, amorphous carbon sheet



**Figure 1:** TEM of carbon nanostructure produced by plasma-assisted conversion of ethanol showing the start of graphene formation on the edge of an amorphous carbon sheet.

## References:

1. M.B. Shavelkina, P.P. Ivanov, R.K. Amirov, A.N. Bocharov, A.I. Drachev, and M.A. Shavelkin, *High Energy Chem.* **55**, 531 (2021).
2. J. Toman, O. Jasek, M. Snirer, V. Kudrle, and J. Jurmanova, *J. Phys. D. Appl. Phys.* **52**, (2019).

# Hydrophobic and Amphiphobic Postmodification of Mesoporous Aerogels via Cold Plasma Coating

B. Schroeter<sup>1</sup>, I. Jung<sup>1</sup>, P. Gurikov<sup>2</sup>, I. Smirnova<sup>1</sup>

<sup>1</sup> Institute for Thermal Separation Processes, Hamburg University of Technology, Hamburg, Germany

<sup>2</sup> Laboratory for Development and Modelling of Novel Nanoporous Materials, Hamburg University of Technology, Hamburg, Germany

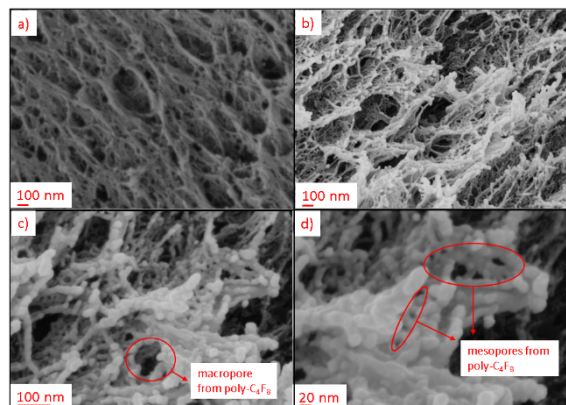
## Abstract:

Aerogels have an enormous potential for a variety of applications due to their special properties such as high pore volumes ( $> 90\%$  v/v), high specific surface areas (up to  $1200 \text{ m}^2/\text{g}$ ) and low densities ( $< 0.2 \text{ g/cm}^3$ ). In particular, their high specific surface areas in combination with the possibility to tailor the pore structures make them suitable as matrices for catalysts, pharmaceuticals or flavors, possibly allowing a targeted and controllable release of the active components. Although, from a material standpoint aerogels offer high variety and potential, the use of aerogels poses challenges in handling and storage: high polarity (in case of polysaccharide gels) and open-porous structure (for all aerogel types) makes biopolymer aerogels especially susceptible to moisture that can penetrate into the pores, resulting in changes of the internal surface and pore collapse. The identification of generally applicable methods for hydrophobization of biopolymer aerogels exterior and internal surface under preservation of the microstructural properties is therefore of particular interest and – so far – a non-solved challenge. Cold plasma coating can be regarded as especially suited for the modification of aerogels, since no liquid solvents are used and the dried aerogels are simply placed in a process chamber and exposed to the glow discharged plasma under moderate vacuum and slightly elevated temperatures (approx.  $40^\circ\text{C}$ ).

In this work, aerogel monoliths with specific surface areas of approx.  $300 \text{ m}^2/\text{g}$  and porosities up to 95% are produced from alginate, cellulose, whey protein isolate (WPI) and potato protein isolate (PPI) and afterwards post-modified via cold plasma coating. Polymerization of different fluorinated monomers (octafluorocyclobutane  $\text{C}_4\text{F}_8$  and perfluoro-acrylates PFAC-6 and PFAC-8) resulted in fast and significant surface hydrophobization after short process times of 5 min and led to (super)hydrophobic exterior surfaces with static water contact angles up to  $154^\circ$ . Simultaneous introduction of hydro- and oleophobicity was possible by deposition of perfluoroacrylates. It was shown, that the porous structure of

aerogels stayed intact during the process. Furthermore, polymerization inside the aerogel pores resulted in generation of new porous moieties and led therefore to a significant increase (up to  $+179 \text{ m}^2/\text{g}$ ) in the specific surface area. The magnitude of the effect depended on the individual process settings and on the overall porosity of the substrates. Summarized, first evidence of cold-plasma induced internal surface modification of biopolymer aerogels is presented, whereas penetration depth of monomer into the pores depended on power input (mode), monomer and substrate porosity and polarity. Additional systematic studies are necessary in order to quantify the influences of power input and input mode from the process side and substrate adhesivity and pore structure from the materials side.

**Keywords:** aerogels, cold plasma coating, mesoporous structure, hydrophobicity, biopolymers



**Figure 1:** Inner pore structure of plasma coated and non-modified alginate aerogels a) pristine aerogel b)- d) coated aerogels, with coating containing intrinsic macro- and mesopores.

## References:

1. Schroeter, B., Jung, I., Bauer, K., Gurikov P., Smirnova, I. (2021) Hydrophobic Modification of Biopolymer Aerogels by Cold Plasma Coating, *Polymers.*, 13, 3000.
2. Smirnova, I., Gurikov, P. (2017), Aerogels in chemical engineering: Strategies toward tailor-made aerogels, *Annu. Rev. Chem. Biomol.*, 8, 307 - 334.

# Plasma Activated Liquids: a Method for Efficient Surface Modification of Semiconductor Nanostructures

P. Galář<sup>1\*</sup>, F. Matějka<sup>1,2</sup>, J. Khun<sup>2</sup> and K. Kůsová<sup>1</sup>

<sup>1</sup>Institute of Physics of the Czech Academy of Sciences, v.v.i, Prague, Czech Republic

<sup>2</sup>University of Chemistry and Technologie in Prague, Prague, Czech Republic

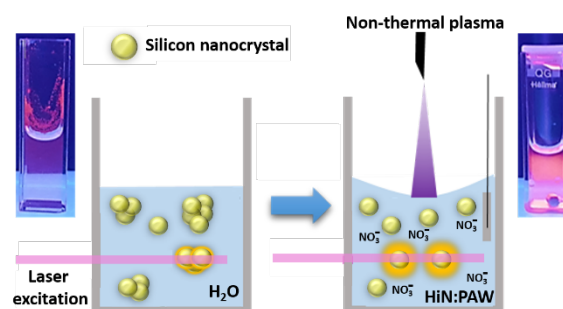
## Abstract:

Nanotechnology represents the cutting edge of the current scientific and also industrial development. The reason of its attractiveness is the possibility to tune significantly properties of bulk materials by limiting their dimensions. An important interesting and environmentally friendly material whose properties still need to be fine-tuned before it can be used commercially are silicon nanocrystals (SiNCs). Beyond standard properties of bulk silicon, the nanocrystals can show relatively high light conversion efficiency with spectrum tunable in the visible spectral region, good stress resistance and also surface reactivity. A wider application of SiNCs is however limited by the absence of a cheap and simple method for their surface termination and modification and poor water dispersibility.

We have recently shown that non-thermal plasma can be effectively used for tuning of the SiNCs surface chemistry through plasma activated liquids (PAL).<sup>1</sup> The technique is based on treating a liquid by an electric discharge, resulting in the rise of reactive species in the liquid. The chemical composition of PAL is dependent on the used atmosphere, discharge properties and type of the liquid. PALs, especially plasma activated water (PAW), were already effectively used in medicine and agriculture. The limiting parameter of common PAW for semiconductor nanotechnology is the presence of a high concentration of hydrogen peroxide that not only damages the nanocrystals, but also blocks any other surface termination besides oxidation. However, we have proven that, using a specific discharge configuration and surroundings, we are able to prepare PAW lacking any hydrogen peroxide and containing a very high concentration of nitrogen radicals (HiN:PAW). The treatment of SiNCs by HiN:PAW resulted in nitrogen-based surface termination, manifested by a significant increase of their quantum yield (more than 10 times) accompanied by an increase of the dispersibility in water (**Figure 1**). The modification is stable for more than a month. While the former effect increases the utilization of SiNCs in light conver-

sion, generation and imaging technology, the latter makes them interesting for further application in hybrid technologies, where water-based chemical processes are involved, such as the preparation of electrodes for lithium batteries. The HiN:PAW treatment was also effectively applied to other types of semiconductor nanoparticles. Moreover, our newer results show that the HiN:PAW modification technique can also be broadened to the application of other PALs based on organic solvents (ethanol, toluene etc.). To successfully achieve plasma activation of these liquids, the construction of the discharge-generating setup has to be again modified.

**Keywords:** Plasma activated liquids, silicon nanocrystals, surface modification, light generation, laser spectroscopy.



**Figure 1:** Simplified behaviour of SiNCs in non-modified water and during surface modification by plasma activated water with simultaneous measurement of their photoluminescence properties. The pictures of cuvettes with SiNCs before and after modification are presented.<sup>1</sup>

## References:

- Galář, P., Khun, J., Fučíková A., Dohnalová, K., Popelář, T., Matulková, I., Valenta, J., Scholtz, V. and Kůsová, K. Non-thermal pulsed plasma activated water: environmentally friendly way for efficient surface modification of semiconductor nanoparticles. *Green Chem.*, 2021, 23, 898-911.



# UV-LED, UV-laser and Corona discharge treatments for polypropylene surface functionalization and optimization of PP-Fiber Reinforced Concrete

B. Malchiodi<sup>1,\*</sup>, P. Pozzi<sup>1</sup>, C. Siligardi<sup>1</sup>

<sup>1</sup> Department of Engineering Enzo Ferrari, University of Modena and Reggio Emilia, Modena, Italy

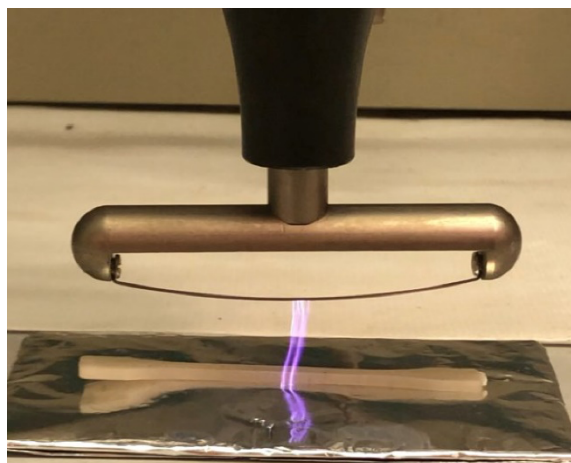
\* Corresponding author: [beatrice.malchiodi@unimore.it](mailto:beatrice.malchiodi@unimore.it)

## Abstract:

Fiber reinforcement is a well-established solution to enhance toughness, prevent the formation and propagation of cracks, thus prevent the early degradation of cement-based products for structural applications. Polypropylene (PP) appears as a valuable fiber reinforcement due to high corrosion resistance, thermal stability, low density and cost. However, its apolar surface strongly affects the compatibility to inorganic matrix; therefore, several surface treatments have been studied, mainly chemical ones, and validated through mechanical testing. In this study, non-chemical surface treatments that might be more sustainable, fast and easy to perform at an industrial scale are investigated: UV-LED, UV-laser and corona discharge treatment. Different exposure times, source to sample distances and setup parameters were considered to optimize treatment effects. Aiming at investigate the effectiveness of surface functionalization between PP and mortar, the treatments were performed on PP plane supports obtained through injection moulding, and validated mainly through chemical-physical properties; i.e. wettability, FTIR, and roughness. The characterization campaign was completed by pullout test of PP plane treated samples from hardened mortar and ESEM-EDX analysis on interphase surface. UV-laser involved both roughness improvements and wettability increase, whereas the increase of UV-LED exposition time up to 36h induced principally a photooxidation. Corona discharge treatment also significantly improved the PP wettability and optimized result were obtained for higher discharge time and distance. The improved surface compatibility between PP and mortar was also confirmed by mechanical and morphological investigations. Consequently, it was demonstrated that the surface treatments involved in this study could positively optimize FRC systems and become sustainable alternative to chemical treatments.

**Keywords:** Fiber Reinforced Concrete, polypropylene, polypropylene fibers, surface

functionalization, compatibility optimization, corona treatment, UV treatment, laser treatment



**Figure 1:** Figure illustrating the corona discharge treatment on a polypropylene plane support to increase its surface functionalization and compatibility optimization to inorganic matrix.

## References:

1. C. Signorini, A. Sola, B. Malchiodi, A. Nobili e A. Gatto, «Failure mechanism of silica coated polypropylene fibres for Fibre Reinforced Concrete (FRC),» *Construction and Building Materiale*, vol. 236, 2020.

# Fast Switch From Hydrophilic to Hydrophobic Surface of Cellulose Film by Low-Temperature Plasma Treatment

A. Oberlintner<sup>1,2,\*</sup>, V. Shvalya<sup>3</sup>, B. Likozar<sup>1</sup>, U. Novak<sup>1</sup>

<sup>1</sup> Department of Catalysis and Chemical Reaction Engineering, National Institute of Chemistry, Ljubljana, Slovenia

<sup>2</sup> International Postgraduate School Jožef Stefan, Ljubljana, Slovenia

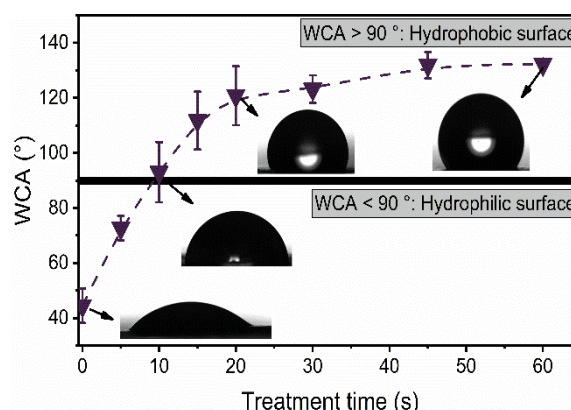
<sup>3</sup> Department of Gaseous Electronics, Jožef Stefan Institute, Ljubljana, Slovenia

## Abstract:

Cellulose is the most abundant natural polymer on the planet and thus is widely available and cheap. Due to its high mechanical strength, non-toxicity, renewability and biodegradability, it is a multifunctional material, suitable for applications in numerous applications. However, its inherent hydrophilicity, a result of abundant hydroxyl groups on the surface, limits its broader use. Various approaches to hydrophobization (esterification, carbamation, silylation, click chemistry) of cellulose have been researched, but these reactions can be time consuming, require harmful solvents and produce liquid discard. Plasma, on the other hand, offers a fast, efficient and stable hydrophobization, producing no waste.

With this in mind, cellulose nanofibrils (CNFs) films were hydrophobized with the means of fluorocarbon (CF<sub>4</sub>) plasma. The surface was transformed from hydrophilic (initial water contact angle (WCA) 46 °) to hydrophobic already in 10 s of treatment. WCA of 129±7° stabilized after 20 s of processing. Furthermore, considering fluorocarbon is a potent greenhouse gas, this study explores utilization of the same plasma system for hydrophobic treatment of cellulose nanomaterials with different dopants, such as nitrogen, carbon and argon as well. The surface modification was carried out at varying processing power and treatment regimes - various lengths of continuous treatment and pulsating processing with intermittent cooling of 5 s. Changes in the structure and morphology of the treated surface was inspected through FTIR, XPS and SEM analyses.

**Keywords:** Cellulose nanomaterials, plasma treatment, hydrophobic, surface modification



**Figure 1:** Water contact angle of CNFs films' surface after CF<sub>4</sub> plasma treatment. The hydrophobicity threshold is reached after 10 s of treatment, while maximum WCA is

## References:

1. Oberlintner, A., Novak, U., Likozar, B. (2021) Hydrophobic functionalization reactions of structured cellulose nanomaterials: Mechanisms, kinetics and in silico multi-scale models. *Carb. Polym.* 259, 117742.
2. Oberlintner, A., Shvalya, V., Vasudevan, A., Vengust, D., Cvelbar, U., Novak, U., Likozar, B. (2022) Hydrophilic to hydrophobic: Ultrafast conversion of cellulose nanofibrils by cold plasma fluorination, *Appl. Surf. Sci.* 581, 152276.
3. Baranov, O., Bazaka, K., Kersten, H., Keidar, M., Cvelbar, U., Xu, S., Levchenko, I., (2017) Plasma under control: Advanced solutions and perspectives for plasma flux management in material treatment and nanosynthesis. *Appl. Phys. Rev.* 4, 041302.



## Recent progress in the electrical management of the plasma electrolytic oxidation process

J. Martin<sup>1,2\*</sup>, V. Ntomproukidis<sup>1,2</sup>, C. Tusch<sup>1</sup>, A. Maizeray<sup>1,2</sup>, G. Marcos<sup>1,2</sup>, T. Czerwicz<sup>1,2</sup>, T. Belmonte<sup>1,2</sup>, G. Henrion<sup>1,2</sup>

<sup>1</sup> Université de Lorraine, CNRS, Institut Jean Lamour, F-54000 Nancy, France

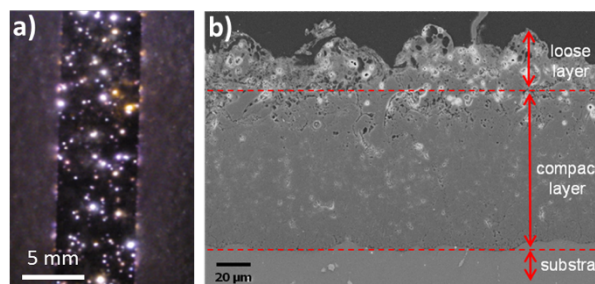
<sup>2</sup> Université de Lorraine, LabEx DAMAS, F-57045 Metz, France

### Abstract:

Plasma electrolytic oxidation (PEO) is a plasma-assisted electrochemical technology to synthesize protective ceramic-like oxide coatings on lightweight metals like aluminium, titanium and magnesium alloys. PEO process is gaining a growing interest in various industrial domains (transport, energy, medicine) to replace conventional acid anodizing processes. Indeed, PEO results in improved wear and corrosion resistance together with the use of environmentally friendly alkaline. PEO process is carried out at a voltage higher than the breakdown voltage of the growing oxide layer. Consequently, PEO coatings grow under a sparking regime leading to the gradual conversion of the processed metal to an oxide layer. The growth mechanisms of the protective PEO coatings remain complex due to the combination of both electrochemical, thermal and plasma phase reactions that simultaneously occur in a small affected volume (tens of  $\mu\text{m}^3$ ) (Figure 1).

After briefly reviewing the main scientific principles of the PEO process including the design of the experimental set-up, the microstructural and functional aspects of the PEO coatings as well as the behaviour of the micro-discharges, the present communication will focus on new achievements into the electrical management of the PEO process. It will be particularly shown that the use of sequential PEO treatments performed by adjusting suitable tuning of the current waveform during the treatment opens opportunities for a better energetic management of the PEO process [1]. The presented results will be discussed based on the relationships drawn between the plasma diagnostic of the micro-discharges, the morphology of the produced PEO coatings and the applied electrical conditions.

**Keywords:** plasma electrolytic oxidation (PEO), micro-arcs oxidation (MAO), Lightweight metal, Aluminium, Magnesium



**Figure 1:** a) Picture of an aluminium sample during the PEO process and b) SEM micrograph cross-section of a PEO processed aluminium sample.

### References:

1. V. Ntomproukidis, J. Martin, A. Nominé, G. Henrion, *Sequential run of the PEO process with various pulsed bipolar current waveforms*, Surface and Coating Technology, 374, 713-724, (2019)

# Promoting resource preservation by PECVD barrier coatings for refillable PET bottles

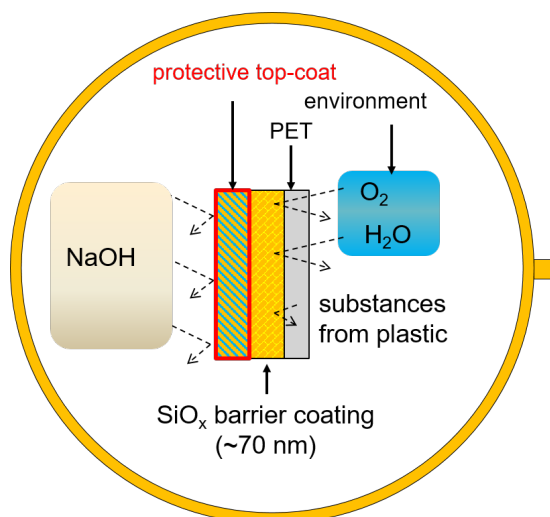
P. Alizadeh<sup>1\*</sup>, R. Dahlmann<sup>1</sup>

<sup>1</sup> Institute for Plastics Processing, RWTH Aachen University, Aachen, Germany

## Abstract:

Alone in the European Union each year around 29.1 mio t of plastic waste accumulated in 2018. 59% of which are directly emitted from plastic packaging. Apart from implied risks to bio diversity and human health due to micro plastics and green house gases, 95 % of packaging material (worth 70 – 105 mio €) are lost to the economy.<sup>1,2</sup> Countries such as Sweden, Denmark, Portugal and Germany have therefore implemented a deposit system i. a. for polyethylene terephthalate (PET) bottles. The German deposit system distinguishes between single use PET bottles and refillable PET bottles.<sup>3</sup> Post-consumer single use PET bottles are shredded into PET flakes and then prepared for reprocessing.<sup>4</sup> Most of the PET-flakes are used to produce new PET bottles (bottle to bottle process). Refillable PET bottles are cleaned and sterilized with NaOH solution after usage and then refilled. Glass bottles can be reused up to 50 times. PET bottles are estimated to be refilled only about 15 times.<sup>5</sup> An ecological assessment of refillable glass bottles, single use PET bottles and refillable PET bottles by the “Institute for Energy and Environmental Research in Heidelberg”, Germany, concluded, that the usage of refillable PET bottles causes the lowest emissions of the greenhouse gas CO<sub>2</sub> in the framework of this study.<sup>6</sup> One important disadvantage of PET is the significantly lower barrier function against gases and thus decreased shelf life compared to glass.<sup>7</sup> Plasma-enhanced chemical vapour deposition (PECVD) is an industrially established method to coat PET bottles on the inside surface with a thin plasma polymeric coating to enhance the bottles' barrier function. For refillable PET bottles, however, this is to date no applicable solution. Conventional barrier coatings are not chemically stable against NaOH solution and are washed away within seconds during the washing process. We developed a protective top-coat, which is applied in the same processing step as the barrier coatings and with the very same base monomers (Hexamethyldisiloxan, Ethin). The coating offers the potential to also store oxygen sensitive products in reusable PET bottles, prevent material odors and prolong the bottles lifecycle. Laboratory tests showed a protective effect over 20 simulated washing cycles. The development is currently being tested for practical application.

**Keywords:** PECVD, thin film development, corrosion protection, food packaging, circular economy, resource preservation.



**Figure 1:** Schematic structure of the developed alkaline resistant PECVD barrier coating for refillable PET bottles

## References:

1. Ellen MacArthur Foundation, *The new plastics economy*, **2016**.
2. Plastics Europe Deutschland e.V., *Plastics – the Facts 2020: An analysis of European plastics production, demand and waste data*, **2020**.
3. Entwurf eines Gesetzes zur Fortentwicklung der haushaltsnahen Getrennterfassung von wertstoffhaltigen Abfällen: Drucksache 18/11274, **2017**.
4. Welle, F. *Resources, Conservation and Recycling* **2011**, 55, 865–875.
5. IFEU - Institut für Energie- und Umweltforschung Heidelberg GmbH. Ökobilanz der Glas- und PET-Mehrwegflaschen der GDB im Vergleich zu PET-Einwegflaschen. Endbericht., **2010**.
6. Palme, A.; Peterson, A.; La Motte, H. de; Theliander, H.; Brelid, H. *Text Cloth Sustain* **2017**, 3.
7. Tinghao F. Wang, T. J. Lin, D. J. Yang, J. A. Antonelli, H. K. Yasuda. *Prog Org Coat* **1996**, 291–297.

# Cathodic plasma electrolytic deposition of an aluminium oxide based hydrogen permeation barrier

Marcel Wetegrove<sup>1\*</sup>, Martin Rohloff<sup>1</sup>, Uwe Lindemann<sup>1</sup>, Antje Quade<sup>1</sup>, Angela Kruth<sup>1</sup>

<sup>1</sup> Leibniz Institute for Plasma Science and Technology (INP), Greifswald, Germany

## Abstract:

Cathodic plasma electrolytic deposition (CPED) is a novel plasma-assisted surface modification technique that enables the formation of ceramic coatings from an electrolytic precursor on conductive metallic substrates such as steel. [1-2] This process represents a highly versatile and application-oriented coating process, as it is applicable to work pieces of complex geometries, can penetrate small gaps and has only a small environmental footprint. In the present work, CPED is used in combination with an electrolyte mixture containing aluminium nitrate and PEG as an additive to deposit oxidic aluminium on stainless steel. During deposition, aluminium nitrate is decomposed in a cathodic plasma discharge. Subsequently, aluminium oxide species are deposited onto the steel substrate due to the high ratio of hydroxyl ions evolved by water reduction at the cathodically polarized metal surface.

The deposition process is optimized towards its potential use for hydrogen barrier coatings. Here, the main objective is to achieve compact layers with as little pores and cracks as possible, showing good adhesion. The layer thickness needs to be adjusted in order to find a compromise between the need for a thin film showing less mechanical stress during pressure and/or temperature cycles, while still delivering a sufficient layer thickness to reduce hydrogen permeation to a tolerable level for the specific substrate and application. This is achieved by tuning the electrical parameters during deposition, additive concentration and the duration of the treatment. The samples are analysed comprehensively by means of electron microscopy, x-ray photoelectron spectroscopy, x-ray diffraction and profilometry to assess the quality of the coatings and to develop a detailed understanding of the processes involved during layer formation.

It is found that the coating quality can be improved significantly by variation of additive concentration, which is known to reduce the influence of the direction of the electric field. The aluminium content varies for different deposition voltages and tends to increase for longer treatment durations. The resulting optimized coating routine delivers homogeneous layers showing

excellent adhesion and is applicable to work pieces of complex shapes.

**Keywords:** cathodic plasma electrolytic deposition, alumina, corrosion protection, hydrogen permeation barrier.



**Figure 1:** Plasma discharges (blue) on the surface of a stainless steel substrate during the cathodic plasma electrolytic deposition process.

## References:

1. Wang, P., Deng, S., He, Y., Liu, C., & Zhang, J. (2016). Influence of polyethylene glycol on cathode plasma electrolytic depositing Al<sub>2</sub>O<sub>3</sub> anti-oxidation coatings. *Ceramics International*, 42(7), 8229-8233
2. Bahadori, E., Javadpour, S., Shariat, M. H., & Mahzoon, F. (2013). Preparation and properties of ceramic Al<sub>2</sub>O<sub>3</sub> coating as TBCs on MCrAlY layer applied on Inconel alloy by cathodic plasma electrolytic deposition. *Surface and Coatings Technology*, 228, S611-S614

## **Plasma Tech Session III: Plasma application for biology, medicine, and agriculture**

# Challenges in Plasma-Conditioned Liquids and Hydrogels for Cancer Treatment

C. Labay<sup>1,2</sup>, F. Tampieri<sup>1,2</sup>, A. Espona<sup>1,2</sup>, X. Solé-Martí<sup>1,2</sup>, M. Mateu-Sanz<sup>1,2</sup>, J. Tornin<sup>1,2</sup>, C. Canal<sup>1,2</sup>

<sup>1</sup> Biomaterials, Biomechanics and Tissue Engineering, Universitat Politècnica de Catalunya (UPC), Barcelona, Spain

<sup>2</sup> Research Centre for Biomedical Engineering (CREB), UPC, 08019 Barcelona, Spain

## Abstract:

The chemistry of Cold Atmospheric Plasma leads to the generation of a variety of reactive oxygen and nitrogen species (RONS) which play a key role in selective cancer cell death without damaging surrounding healthy tissues. This has led to advancing research on plasmas in a variety of cancer types. Taking advantage of the reactivity of plasmas in liquids, plasma-conditioned liquids (PCL) can be generated and represent a very interesting alternative to direct CAP treatment because they open the door to minimally invasive therapies [1] [2].

In this work we will focus in a kind of cancer of pediatric prevalence, Osteosarcoma (OS). OS is the most common bone malignant neoplasm. Although chemotherapy has improved long-term survival over the past few decades, the outcome for patients with metastatic or recurrent OS remains dismally poor.

The use of PCL can be a useful tool in the treatment of this malignancy [3], and we have been investigating the generation of RONS in different biocompatible liquids, ranging from saline solutions to more complex biopolymer-containing solutions. In this talk we will discuss some of our investigations on the nature and extent of the modifications induced by CAP treatment on biopolymer solutions. The results allowed observing that the concentration of biopolymers affects the concentration of RONS detected in the liquid phase, pointing out direct interactions between RONS and biopolymer chains. The use of molecular models helped us to identify the outcomes of these interactions, thus shedding light on the chemical mechanisms involved.

The RONS generated in different solutions and hydrogels will be discussed in relation to their biological effects, as investigated in 2D monolayer cultures as well as in new 3D-OS tumor model specifically developed to study PCL as anti-tumoral agent [4], and effects in vivo will also be discussed.

**Keywords:** cold atmospheric plasma, plasma conditioned liquids, hydrogels, reactive oxygen and nitrogen species.

## References:

1. M. Keidar, A. Shashurin, O. Volotskova, et al., (2013). Cold atmospheric plasma in cancer therapy. *Physics of Plasmas*, 20, 057101.
2. A. Khlyustova, C. Labay, Z. Machala, M.-P. Ginebra, C. Canal (2019). Important parameters in plasma jets for the production of reactive species in liquids for plasma medicine: a brief review. *Front. Chem. Sci. Eng.* 13, 238–252.
3. M. Mateu-Sanz, J. Tornin, B. Brulin, A. Khlyustova, M.-P. Ginebra, P. Layrolle, C. Canal (2020). Cold plasma-treated Ringer's Saline: A weapon to target Osteosarcoma. *Cancers* 12, 227.

## Acknowledgments

Authors acknowledge the European Research Council (ERC) under the European Union's Horizon 2020 research and innovation programme (Grant agreement N° 714793) and Cost Action 20114 PlasTHER. MINECO for PID2019-103892RB-I00/AEI/10.13039/501100011033 project. Authors acknowledge Generalitat de Catalunya for SGR2017 1165 and for the Icrea Academia award of CC.



# Multifunctional medical materials of the future by plasma surface modification

I. Junkar <sup>1\*</sup>, M. Benčina <sup>1</sup>

<sup>1</sup> Department of surface engineering, Jožef Stefan Institute, Ljubljana, Slovenia

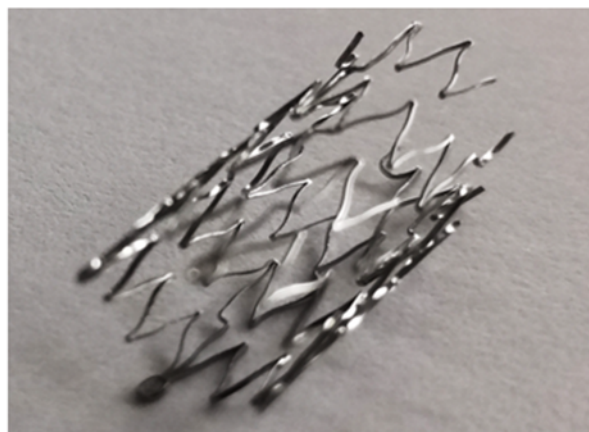
## Abstract:

With rapid aging of the global population and the rise of antibiotic-resistant bacterial strains, there is an increased demand to design biomaterial surfaces with superior multifunctional properties. It is now a well known fact that cell interaction is dictated also by surface nanotopography [1] and that by fine tuning of surface features desired biological response could be achieved. Combining novel approaches for nanostructuring of metal biomaterials, like electrochemical anodization or hydrothermal treatment in combination with plasma treatment technologies could offer new possibilities for fabrication of surfaces with multifunctional properties.

Surfaces of implantable materials could be specifically designed to provide selective cell adhesion (promote adhesion of one cell type over another) and at the same time prevent bacterial infections (biofilm formation). Plasma technologies provide interesting approach for surface modification of medical implants, as surface features like wettability, chemistry, nanotopography and crystallinity can be modified without influencing the bulk attributes of the material. By appropriately tuning surface features, multifunctional surfaces with desired biological response for specific medical application could be attained. As designing of medical device surface is highly challenging, usually achieving optimal surface properties is a trade-off. Thus preparation of surfaces with multiple functionalities is even harder to attain.

Approaches for fabrication of nanostructured surfaces for vascular and orthopaedic implants will be presented and discussed in more detail. It will be shown that gaseous plasma treatment at optimal treatment conditions provides selective cell response on nitinol vascular stent surface. Example of nitinol vascular stent is presented in Figure 1. As vascular stent surfaces still lack of desired biological response due to high risk for restenosis and thrombosis, solutions to overcome these issues are intensively sought. By combining hydrogen and oxygen plasma at specific treatment conditions, multifunctional properties of nitinol vascular stent could be achieved. Treatment enables good proliferation of endothelial cells that line our natural blood vessels and are

thought to be an ideal antithrombogenic material, while proliferation of smooth muscle cells (issues with restenosis) and adhesion of platelets (issues with thrombosis) are significantly reduced. Combination of other surface nanostructuring techniques and plasma [2] will also be presented and discussed in the light of vascular stents as well as orthopaedic implants.



**Figure 1. Nitinol vascular stent, kindly donated by Rontis.**

**Keywords:** low pressure plasma, vascular stents, orthopedic implants, biocompatibility, nanostructure, biomedical applications.

## References:

1. Chen, C., S. Bang, Y. Cho, S. Lee, I. Lee, S. Zhang and I. Noh (2016). "Research trends in biomimetic medical materials for tissue engineering: 3D bioprinting, surface modification, nano/micro-technology and clinical aspects in tissue engineering of cartilage and bone." *Biomaterials research* 20 (1): 1-7
2. Benčina, M., N. Rawat, K. Lakota, S. Sodin-Šemrl, A. Iglič and I. Junkar (2021). "Bio-Performance of Hydrothermally and Plasma-Treated Titanium: The New Generation of Vascular Stents." *Int. J. Mol. Sci.* 22(21): 11858.



# Improved biocompatibility of hydrothermally and plasma-treated titanium

Metka Benčina<sup>1,2</sup>, Niharika Rawat<sup>2</sup>, Pia Starič<sup>1</sup>, Katja Lakota<sup>3</sup>, Snežna Sodin-Šemrl<sup>3</sup>, Aleš Iglič<sup>2,4</sup>, Ita Junkar<sup>1</sup>

1 Department of Surface Engineering, Jožef Stefan Institute, Jamova 39, SI-1000, Ljubljana, Slovenia

2 Laboratory of Physics, Faculty of Electrical Engineering, University of Ljubljana, Tržaška 25, SI-1000 Ljubljana, Slovenia

3 Department of Rheumatology, University Medical Centre Ljubljana, Vodnikova 62, SI-1000 Ljubljana, Slovenia

4 Faculty of Medicine, University of Ljubljana, Vrazov trg 2, SI-1000, Ljubljana, Slovenia

## Abstract:

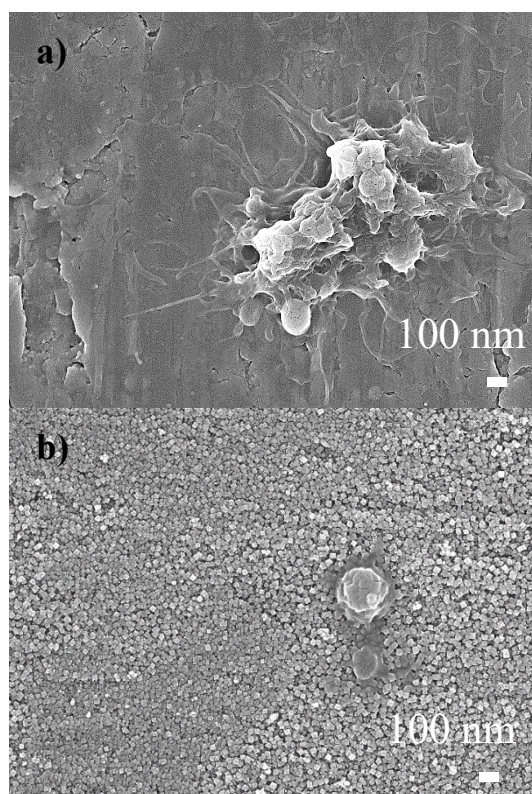
Cardiovascular diseases (CVD) account for 45% of all deaths in Europe (4 million people) [1] and the rate of mortality associated with CVD is continuously rising. The most common form of heart disease, coronary artery disease (CAD), is the buildup of plaque inside the blood vessels. Implantation of vascular stents, usually made of metallic alloys, are common treatment for patients with CAD, used to open narrowed vessel and restore blood flow. Although titanium alloys are extensively used for vascular stent application, they still lack of desired biological responses.

In present study, titanium substrate has been subjected to hydrothermal treatment (HT) followed by treatment with reactive oxygen species (non-thermal oxygen plasma - P) [1]. Samples were characterized by scanning electron microscopy (SEM) with energy-dispersive X-ray analysis (EDX), atomic force microscopy (AFM), X-ray photoelectron spectroscopy (XPS) and water contact angle (WCA) analysis.

The combination of hydrothermal and plasma treatment resulted in the formation of an oxygen-rich nanostructured titanium oxide layer. The alteration of surface properties, such as morphology, surface roughness, elemental composition and wettability influenced the material's bio-performance; platelet adhesion and aggregation was reduced on surfaces treated by hydrothermal treatment, as well as after plasma treatment. It has been also shown that surfaces treated by both treatment procedures (HT and P) promoted the adhesion and proliferation of vascular endothelial cells, while at the same time inhibited the adhesion and proliferation of vascular smooth muscle cells.

The nanostructurization, combined with non-thermal plasma treatment, which altered physico-chemical properties of the titanium, enabled the formation of a surface with characteristics, especially appropriate for use as vascular stents.

**Keywords:** titanium, medical implants, nanostructured surface, hydrothermal method, non-thermal plasma.



SEM images of a.) Ti foil treated with plasma (Ti+P), and (b) hydrothermally/plasma treated Ti foil (Ti HT+P) after incubation with whole blood. Adapted from Ref [2].

## References:

1. MOVSISYAN, Narine K., et al. Cardiovascular diseases in Central and Eastern Europe: A call for more surveillance and evidence-based health promotion. *Annals of global health*, 2020, 86.1.
2. BENČINA, Metka, et al. Bio-Performance of Hydrothermally and Plasma-Treated Titanium: The New Generation of Vascular Stents. *International Journal of Molecular Sciences*, 2021, 22.21: 11858.

# PECVD coatings as migration barriers for the use of post-consumer recyclates in food contact

L. Kleines<sup>1\*</sup>, R. Dahlmann<sup>1</sup>

<sup>1</sup> Insitute for Plastics Processing, RWTH Aachen University, Aachen, Germany

## Abstract:

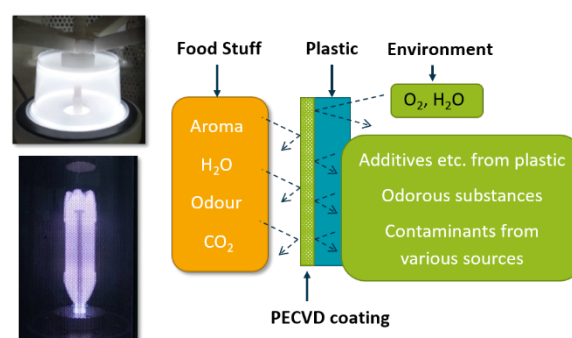
The European Commission's Plastics Strategy, published in January 2018, sets the goal to recycle at least half of the plastic waste by 2030 and thus to substantially increase the share of recyclates used in newly produced plastic products.<sup>1</sup> Although the packaging sectors demand in Europe has the highest share (almost 40%) compared to other sectors<sup>2</sup>, the success of the European initiative to increase the use of recyclates is currently hindered by, among other things, the very limited possibility of using recycled plastics in the food sector. One of the main reasons for this is the risk of migration of chemical substances from the used packaging into the food and a subsequent lack of safety of the food contact materials evaluated in an assessment of safety by the European Food Safety Authority (EFSA). Sufficient product protection in terms of toxicity, taste and odour changes as well as contamination of various kinds cannot be guaranteed for most materials especially when using post-consumer recyclates (PCR) other than polyethylene terephthalate (PET).

One solution to this problem is to explore and further develop the potential of highly functional PECVD (plasma enhanced chemical vapour deposition) coating systems as migration barriers for contaminants from recyclates. Thereby we focus on non-PET materials such as polypropylene (PP), as this is where the biggest hurdles lie for the use of recyclates in food contact. Basic research and at the same time application-oriented experiments and theoretical models are being used to understand different effects and processes in the system consisting of recycled plastic, the PECVD coating and a foodstuff. The use of functional migration barriers in this case not only has a high level of potential from a process technological perspective, but also from a regulatory point of view, as EU regulations declare that behind a functional barrier, non-authorized substances may be used, provided they fulfil certain criteria and their migration remains below a given detection limit.

Methodologically, in a first step, test substances are used to simulate the contamination of the recyclates and their uptake by food. Suitable coat-

ing systems are then developed on these contaminated materials, which requires intensive analysis due to the unknown surface properties of the recyclates. These coating systems are tested in extensive migration analytical studies for their suitability for migration reduction.

**Keywords:** migration barriers, PECVD, post-consumer recyclates, food packaging, thin film development, EU plastics strategy, circular economy



**Figure 1:** Using PECVD coatings as migration barriers for contaminated recyclates in food contact.

## References:

1. European Commission (2018), A European Strategy for Plastics in a Circular Economy, *Communication from the Commission to the European Parliament*.
2. PlasticsEurope (2020), Plastics – the Facts 2020: An analysis of European plastics production, demand and waste data.
3. European Commission Regulation (EU) No 10/2011 (2011), Regulation on plastic materials and articles intended to come into contact with food.

# Utilization of Plasma in Medicine: Inactivation of *Acanthamoebas*, Dermatophytes, and Staphylococci

T. Měřínská<sup>1</sup>, E. Lokajová<sup>1</sup>, J. Julák<sup>2</sup>, E. Nohýnková<sup>2</sup>, J. Khun<sup>1</sup>, V. Scholtz<sup>1</sup>, P. Petráš<sup>3</sup>

<sup>1</sup> Department of Physics and Measurements, University of Chemistry and Technology, Prague, Czech Republic

<sup>2</sup> Institute of Immunology and Microbiology, 1st Faculty of Medicine, Charles University and General University Hospital, Prague, Czech Republic

<sup>3</sup> National Reference Laboratory for Staphylococci, National Institute of Public Health, Prague, Czech Republic

## Abstract:

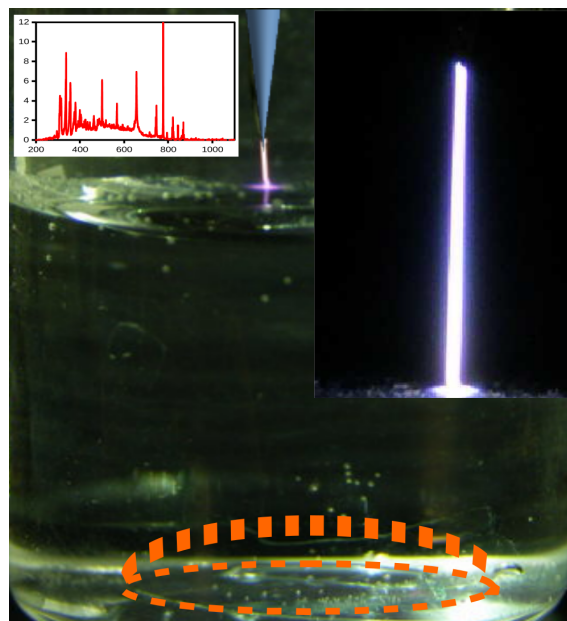
Non-thermal plasma has been successfully used in a wide range of medical fields in recent years. It is widely used not only for the microbial inactivation or disinfection, but also in other areas of medicine. Here, we present three our new insights in plasma medicine related to disinfection: the disinfection of *Acanthamoeba* sp. [1], dermatophytes [2] and staphylococci.

*Acanthamoeba* is the causative agent of infectious eye disease in humans. In its life cycle, it has a stage of dormant cysts that is very resistant to disinfection. In our research, the cysts were inactivated by the DC corona transient spark discharge both in suspension or placed on the surface of contact lenses. The negative discharge appeared to be more effective than the positive one. The complete inactivation occurred in water suspension after 40 min and on contaminated lenses after 50 min of plasma exposure. The properties of lenses seem to not be affected by plasma exposure; that is, their optical power, diameter, curvature, water content, and infrared and Raman spectra remain unchanged.

In a study of the therapy of onychomycosis, we monitored the dermatophytes sensitivity to non-thermal plasma generated by DC corona discharge in the point-to-ring arrangement. We used various strains of *Trichophyton interdigitale*, *Trichophyton benhamiae*, *Trichophyton rubrum*, and *Microsporum canis*. All strains were sensitive to plasma action, but the sensitivity and inactivation dynamics vary from strain to strain and is not an inherent property of the fungal species. The studied strains can be divided into four types according to their growth trends observed after plasma exposure as follows: The “Strong effect” type displayed almost complete growth suppression after exposure. In contrast, the “No effect” included strains whose growth was not inhibited at all. The type “Soft effect” included strains whose growth was only slightly inhibited. In the “Kick off effect” type, an initial slowdown of growth, followed by a rapid growth, was observed.

*Staphylococcus aureus* from clinical material (especially abscesses and wounds) was also exposed to plasma in water suspension and on agar plates. The results show that staphylococci are sensitive to plasma after a few minutes of exposure.

**Keywords:** *Acanthamoeba*, cysts, contact lenses, corona discharge, IR spectra, Raman spectra, *Microsporum*, treatment of mycoses, *Trichophyton*



**Figure 1:** Contact lens exposure.

## References:

1. Měřínská, T., Scholtz, V., Khun, J., Julák, J., Nohýnková, E. (2021), Inactivation of *Acanthamoeba* Cysts in Suspension and on Contaminated Contact Lenses Using Non-Thermal Plasma, *Microorganisms*, 9, 1879.
2. Lokajová, E., Julák, J., Khun, J., Soušková, H., Dobiáš, R., Lux, J., Scholtz, V. (2021), Inactivation of Dermatophytes Causing Onychomycosis Using Non-Thermal Plasma as a Prerequisite for Therapy, *J. Fungi*, 7, 715.

## Understanding the molecular mechanisms of non-thermal plasma treatments on *Arabidopsis thaliana* seeds

A. Waskow<sup>1</sup>, A. Guihur<sup>2</sup>, A.A. Howling<sup>1</sup>, I. Furno<sup>1</sup>

<sup>1</sup>École Polytechnique Fédérale de Lausanne, Swiss Plasma Center, Lausanne, Switzerland

<sup>2</sup>University of Lausanne, Department of Plant Molecular Biology, Lausanne, Switzerland

### Abstract:

Plasma agriculture is a rapidly emerging interdisciplinary field where non-thermal plasmas are used to treat heat-sensitive biological substrates, such as seeds and plants. When dosed adequately, plasma-treated seeds are observed to have accelerated germination, enhanced growth, reduced water consumption, increased crop yield and disease resistance, and decreased levels of microbial pathogens.

Currently, more is known about how non-thermal plasma influences the macroscopic properties of plants but little is known about how plants are affected on a molecular level. Furthermore, it remains unclear which component (reactive oxygen and nitrogen species (RONS), heat, electric or magnetic fields, UV, ions, electrons) are responsible for an observed plasma effect on seeds and their subsequent development. Here, we use Surface Dielectric Barrier Discharge (SDBD) treatments of the model plant organism *Arabidopsis thaliana* Columbia-0 to investigate the underlying molecular effects of plasma treatments.

Plant seeds were exposed to DBD plasmas, to identify conditions yielding a phenotypic change, specifically accelerated germination. Variables during the treatment, such as exposure time, distance, voltage waveform, flow rate are considered, revealing that AC-powered DBD with particular time and voltage can hasten germination. Although it is unclear which plasma component is responsible for this effect, mRNA was extracted from plasma-treated seeds 6 days after sowing and revealed that the plasma treatment did indeed change the molecular program in the plant and upregulated the defense pathways. The type of defense response was dependent on plasma time exposure. Lastly, guidelines about protocol recording are shared in an effort to synchronize efforts in plasma agriculture to understand which parameters are important for designing plasma-seed treatments to ultimately transfer this technology into industry.

**Keywords:** non-thermal plasma, *Arabidopsis*, DBD, germination, seeds, molecular biology, RNA sequencing, transcriptomics

### References:

1. Waskow, A., Guihur, A., Howling, A., Furno, I. (2022) RNA sequencing of plasma-treated *Arabidopsis thaliana* seeds reveals upregulation in plant stress and defense pathways. Manuscript in preparation.
2. Waskow, A., Howling, A., Furno, I. (2021) Mechanisms of plasma-seed treatments as a potential seed processing technology. *Front. Phys.* 9:617345.
3. Waskow, A., Avino, F., Howling, A., Furno, I. (2021). Entering the plasma agriculture field: An attempt to standardize protocols for plasma treatment of seeds. *Plasma Processes and Polymers*, e2100152.



# Inactivation of Critically Ranked Carbapenem Resistant Bacteria and Genes Using Cold Atmospheric Plasma Technology

Thabang B.M. Mosaka<sup>1</sup>, Michael O. Daramola<sup>1</sup>, Chedly Tizaoui<sup>2</sup>, Samuel A. Iwarere<sup>1\*</sup>

<sup>1</sup> Department of Chemical Engineering, University of Pretoria, City, South Africa

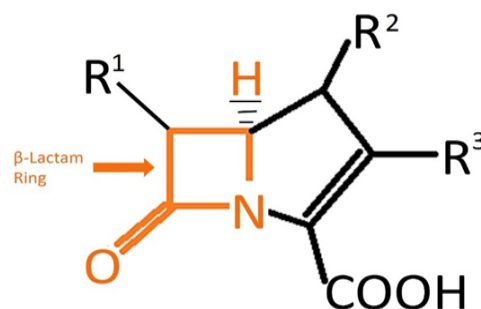
<sup>2</sup> College of Engineering, Swansea University, Swansea SA1 8EN, United Kingdom

## Abstract:

Antibiotics are used for control of bacterial infections in humans and animals, for cancer treatment and as growth promotion agents (Sarangapani et al., 2019, Yuan et al., 2015). The most used antibiotics worldwide are Beta-lactams ( $\beta$ -lactams) which include penicillins, cephalosporins, monobactams and carbapenems. The common factor among them is a  $\beta$ -lactam ring which they all share and they act the same by binding to and inactivating the penicillin-binding proteins (PBPs). The PBPs are responsible for the formation of the bacterial cell wall. (Meletis, 2016). Among the  $\beta$ -lactams, carbapenems are considered to be the most reliable last-resort for treating bacterial infections as they are the more effective against Gram-negative and Gram-positive bacteria (Figure 1) (Meletis, 2016, Codjoe and Donkor, 2017).

Wastewater treatment plants are not equipped for the removal of antibiotics and degradation of antibiotic-resistant bacteria (ARB) and antibiotic resistance genes (ARGs) which have been reported in treated drinking water, river water, soil, and even air (Sarangapani et al., 2019, Yuan et al., 2015). The World Health Organisation (WHO) ranked the ARBs; *Acinetobacter baumannii*, carbapenem-resistant and *Pseudomonas aeruginosa*, carbapenem-resistant as priority 1 (critical) because they pose a particular threat (World Health Organisation, 2017). This threat is evident as patients with infections caused by the critically ranked bacteria consume more health-care resources, because they are more at risk of worse clinical outcomes and death than patients infected with non-resistant strains of the same bacteria (World Health Organisation, 2020). The aim of this study is to investigate plasma-based technology as a disinfection method that produces  $OH$  and other discharges, to not only inactivate all bacteria but also inactivate their genes, thereby eliminating antibiotic resistance.

**Keywords:** carbapenem, antibiotic resistant bacteria, antibiotic resistant genes, disinfection, cold atmospheric plasma.



**Figure 1:** Molecular Structure of a Carbapenem

## References:

1. Codjoe, F. S., Donkor, E. S. (2017), Carbapenem Resistance: A Review. *Med Sci (Basel)*, 6, 1-28.
2. Meletis, G. (2016), Carbapenem resistance: overview of the problem and future perspectives. *Therapeutic advances in infectious disease*, 3, 15-21.
3. Sarangapani, C., Ziuzina, D., Behan, P., Boehm, D., Gilmore, B., Cullen, P. J., Bourke, P. (2019), Degradation kinetics of cold plasma-treated antibiotics and their antimicrobial activity. *Scientific Reports*, 9(3955), 1-15
4. World Health Organisation. (2017), WHO publishes list of bacteria for which new antibiotics are urgently needed [Online]. Available: <https://www.who.int/news/item/27-02-2017-who-publishes-list-of-bacteria-for-which-new-antibiotics-are-urgently-needed> [Accessed 2021].
5. World Health Organisation. (2020), Antimicrobial resistance [Online]. Available: <https://www.who.int/en/news-room/fact-sheets/detail/antimicrobial-resistance> [Accessed 2021].
6. Yuan, Q. B., Guo, M. T., Yang, J. (2015), Fate of Antibiotic Resistant Bacteria and Genes during Wastewater Chlorination: Implication for Antibiotic Resistance Control. *PLOS ONE*, 10(e0119403), 1-11.

**SICT 2022 / Tribology 2022 Joint  
Session I:  
Coatings and Surfaces  
Corrosion / tribological  
properties / adhesion and  
Adhesives**



# Corrosion Protection Potential of Hard Nitride Based Coatings Produced with PVD techniques

M. Ürgen<sup>1\*</sup>, B. Avci<sup>1</sup>

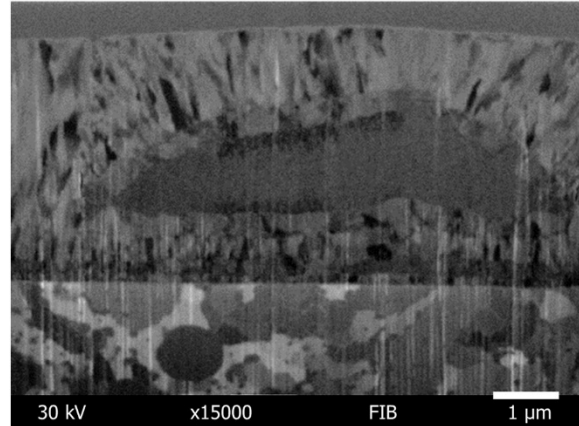
<sup>1</sup> Department of Metallurgical and Materials Engineering, Istanbul Technical University, Istanbul, Turkey

## Abstract:

Nitride based coatings, such as TiN, TiAlN and CrN are widely used in industrial applications. Their high chemical resistance, hardness and wear resistance makes them ideal coatings for a wide range of applications such as cutting and forming tools, automotive, aerospace and machine components, biomaterials. Additionally, they are also used as decorative coatings due to their appealing colors and scratch resistance. The high self corrosion resistance of these coatings also makes them potential candidates for corrosion protection. However, the presence of defects, mainly arising from their columnar nature, that extend to the substrate considerably restrict their applications for corrosion protection (Figure 1). Another widely accepted mechanism for accelerated corrosion in the presence of these coatings is the galvanic interactions between the substrate and the coating. There are many studies in the literature on the improvement of the corrosion protection ability of these coatings. These studies are concentrated on changing the morphology of the coatings, interface or substrate.

Within the scope of this study, after giving an overview on the corrosion of hard ceramic coated materials, strategies for improvement of corrosion protection ability will be summarized. The possible contribution of galvanic interaction of these coatings with different substrate materials to the corrosion of these systems will be specifically stressed and recent experimental results on this topic will be presented.

**Keywords:** corrosion protection, PVD coatings, hard nitride based coatings, defect, macroparticles, galvanic corrosion.



**Figure 1:** The inherent defective nature of coatings affect the corrosion protective ability of the coatings by providing an electrolytic pathway between substrate and coating.

## References:

1. Akkaya, S.S., Sireli, E. Alkan, B., Kazmanli, M.K., Ürgen, M. (2011). Effect of cathodic arc plasma treatment on the properties of WC-Co based hard metals, *Surface and Coatings Technology*, 206 (7), 1759-1764.
2. Azar, G.T.P., Yelkarasi, C., Ürgen, M. (2017). The role of droplets on the cavitation erosion damage of TiN coatings produced with cathodic arc physical vapor deposition, *Surface and Coatings Technology*, 322, 211-217.
3. Er, D., Azar, G.T.P., Kazmanli, K., Ürgen, M. (2018). The corrosion protection ability of TiAlN coatings produced with CA-PVD under superimposed pulse bias, *Surface and Coatings Technology*, 346, 1-8.

# Anticorrosive PEO coatings on metallic cast heat enhancers for thermal energy storage

N. Rażny<sup>1\*</sup>, A. Dmitruk, K. Naplocha

<sup>1</sup>Department of Lightweight Elements Engineering, Foundry and Automation, Wrocław University of Science and Technology, Wrocław, Poland

## Abstract:

The increasing heat demand inflicted an intensive development in heat storage techniques. Thermal energy storage (TES) units use phase change materials (PCM), characterized by high enthalpy of phase transition, to accumulate a large amount of energy. During heating (charging) the PCM melts and absorbs heat, and releases it while cooling down (discharging). Eutectic salt mixtures and salt hydrates ( $\text{KNO}_3\text{-NaNO}_3$ ,  $\text{MgCl}_2\cdot 6\text{H}_2\text{O}$ ,  $\text{LiNO}_3\text{-KNO}_3\text{-NaNO}_3$ , etc.) are often used for this purpose (enthalpies: 100 J/g, 170 J/g, 168 J/g, respectively) [1–3]. Such materials, apart from high enthalpy, feature low thermal conductivity. To overcome this issue, cast metallic spatial structures, often called inserts, produced via combined methods of additive manufacturing and investment casting are proposed [4,5].

In molten salt hydrates environment, at elevated temperatures, metal inserts are exposed to aggressive, highly corrosive conditions, which may adversely affect their quality and lifetime. For this reason, the use of ceramic coatings obtained by plasma electrolytic oxidation (PEO) is tested. PEO is a method of coating lightweight metal alloys with oxide layers under the influence of high voltage [6].

In the proposed work, the effect of different PEO parameters (concentration of electrolyte solution, duration of the process, voltage) on the quality of the layers obtained on aluminum alloy (AC 44200) is investigated. Scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS), X-ray diffraction (XRD) are utilized to examine morphology and microstructure of produced PEO coating. To evaluate corrosion resistance, the potentiodynamic test, as well as long-term exposure to molten salts trials, are run. Aluminum alloy and PEO coated samples are subjected to molten  $\text{MgCl}_2\cdot 6\text{H}_2\text{O}$  environment at the temperature of 130°C for different periods. Corrosion products are defined. The best performing coating is chosen to be tested in a lab-scale PCM deposit and the charging-discharging (heating-cooling) curves are recorded.

The research shows that the coating does not affect charging and discharging process

significantly, while the lifetime of the structures is notably prolonged.

**Keywords:** Thermal energy storage, plasma electrolytic oxidation, PEO, aluminum, corrosion, Phase Change Materials, PCM, heat storage, salt hydrates

## References:

1. Bauer, T., Laing, D., and Tamme, R. (2010) Overview of PCMs for Concentrated Solar Power in the Temperature Range 200 to 350°C. in: AST, pp. 272–277.
2. D’Aguzzo, B., Karthik, M., Grace, A.N., and Floris, A. (2018) Thermostatic properties of nitrate molten salts and their solar and eutectic mixtures. *Scientific Reports*. 8 (1), 10485.
3. Ahmed, F. and Waqas, A. (2019) Experimental Investigation Of Using Latent Thermal Energy Storage System Comprising Of Magnesium Chloride Hexahydrate ( $\text{MgCl}_2\cdot 6\text{H}_2\text{O}$ ) With Domestic Gas Heater. in: 2019 3rd Int. Conf. Energy Conserv. Effic., IEEE, pp. 1–6.
4. Naplocha, K., Dmitruk, A., Grzęda, J., and Kaczmar, J. (2021) The effect of an Al-based cellular structure on the thermal performance of a zeolite-based hybrid heat accumulator. *International Communications in Heat and Mass Transfer*. 129 105724.
5. Dmitruk, A., Naplocha, K., Grzęda, J., and Kaczmar, J.W. (2020) Aluminum inserts for enhancing heat transfer in PCM accumulator. *Materials*. 13 (2), 415.
6. Sobolev, A., Kossenko, A., Zinigrad, M., and Borodianskiy, K. (2018) Comparison of plasma electrolytic oxidation coatings on Al alloy created in aqueous solution and molten salt electrolytes. *Surface and Coatings Technology*. 344 590–595.

# Self-healing core-shell nanofibres for corrosion protective coatings

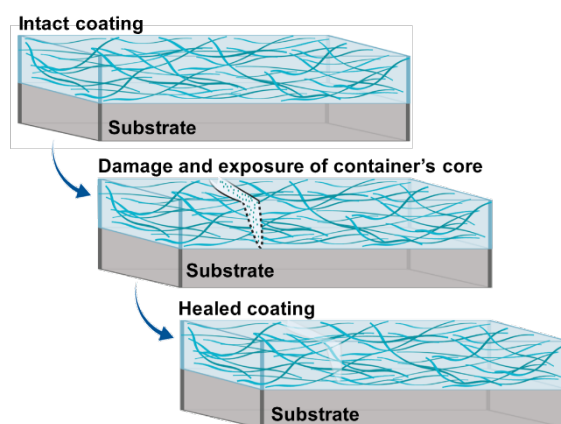
N. C. M. Spera\*, J. P. S. Sousa

Iberian Nanotechnology Laboratory (INL International), Braga, Portugal

## Abstract:

Protective coatings are applied to ensure durability and easy maintenance of structures in order to reduce failures, malfunctioning and minimize costs. For this application, Self-healing materials are distinguished by the ability to recover its properties and functionality after a damage. When applied in a coating, the healing agent can be intrinsic from the polymer and activated by an external source of energy or stored in a container until it receives a stimulus to act and autonomously respond to it. For corrosion protective coatings, the approach using nanocontainers are the most promising ones, specially nanofibres due to its aleatory orientation and entangled, inter-connected, three-dimensional network. We produced core-shell nanofibres using coaxial electrospinning, in which the core contains the healing agent. Based on silyl ester, our mechanism does not require a secondary stimulus, once the damage occurs, it cut the fibres and the core is exposed to humidity. By hydrolysis, it creates a hydrophobic densified layer between the metal and the corrosive atmosphere, protecting the substrate (Figure 1). The nanofibers were characterized by Fourier-transform infrared spectroscopy (FTIR), Scanning electron microscope (SEM) and Thermogravimetric Analysis (TGA). To be applied in a coating formulation, the nanofibers were dispersed in a solvent and spray coated with a polydimethyl-siloxane polymer (PDMS) matrix. The protective behaviour and the healing ability of the final coating were investigated by Electrochemical Impedance Spectroscopy (EIS) with a three electrode system and sea water media. The self-healing effect was evaluated after a forced mechanical trigger and exposition to humidity, during different time. The results obtained so far intensified the potential of our core-shell nanofibres as an advanced material to be used in coatings in order to increase their durability and protective properties.

**Keywords:** self-healing materials, self-healing coatings, core-shell fibres, anticorrosive coatings, advanced protective coating, electrochemical impedance spectroscopy.



**Figure 1:** Figure illustrating the three-dimensional network formed by the fibres dispersed at the coating layer (intact coating). The self-healing activity is also exposed, considering the mechanical damage as a trigger to initiate the process.

## References:

1. Hansen, C. J., Wu, W., Toohey, K. S., Sottos, N. R., White, S. R., Lewis, J. A. (2009) Self-Healing Materials with Interpenetrating Microvascular Networks, *Adv. Mater.*, 21, 4143–4147.
2. An, S., Liou, M., Song, K. Y., Jo, H. S., Lee, M. W. Al-Deyab, S. S., Yarin, A. L., Yoon, S. S. (2015), Highly flexible transparent self-healing composite based on electrospun core-shell nanofibers produced by coaxial electrospinning for anti-corrosion and electrical insulation, *Nanoscale*, 7, 17778–17785.

# Epoxy Novolac Coating Interactions at High Pressure High Temperature Conditions

N. Rajagopalan<sup>1,\*</sup>, C.E. Weinell<sup>1</sup>, K. Dam-Johansen<sup>1</sup>, S. Kiil<sup>1</sup>

<sup>1</sup> CoaST, Chemical Engineering Department, Technical University of Denmark (DTU), Denmark

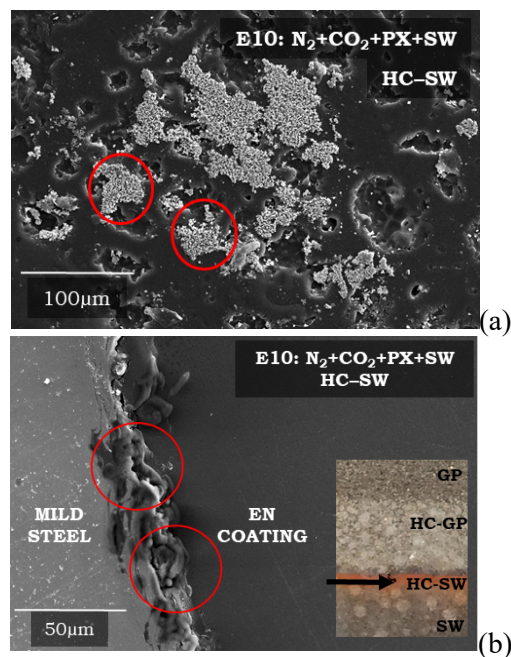
## Abstract:

Despite the progress in the usage of renewable resources, the oil and gas industries rely increasingly on wells and reservoirs operating at high pressure and high temperature (HPHT) conditions [1]. Under such conditions, and in the presence of hydrocarbons, seawater, and CO<sub>2</sub>, the substrate (steel) corrosion rate increases, along with the risk of mechanical failures. To avoid material degradation at HPHT, process equipment, transportation pipelines, and underground storage tanks are normally coated internally with high performance, epoxy-based coating systems. Owing to the complex and multi-phase situation at HPHT, substrate corrosion and coating degradation cannot, at present, be prevented, and the underlying degradation mechanisms remain more or less unknown.

Using an exclusive three-phase batch reactor that simulate the HPHT conditions [2], this investigation studies the influence of carbon dioxide (CO<sub>2</sub>), present in the gas phase at conditions of HPHT, on the degradation and surface interactions of an amine-cured epoxy novolac (EN) coating. In addition, how the hydrocarbon associated surface softening (glass transition temperature depression) allowed the dissolved CO<sub>2</sub> gas to diffuse into the EN network is explained. In what way the synergistic action of CO<sub>2</sub>, para-xylene, and seawater resulted in an increased chain motion of the EN network, subsequently allowing CO<sub>2</sub> and seawater ions to diffuse the EN crosslinking to the steel substrate, imposing underfilm corrosion (Figure 1b) is mapped and discussed. Lastly, the rapid gas decompression (RGD), i.e. the depressurization to ambient conditions, and its influence on the deposition of underfilm corrosion products on the coating surface (Figure 1a) is described.

The findings, additionally, have the potential to provide a muchneeded supercritical CO<sub>2</sub>-resistant epoxy-based candidate to protect transport pipelines for the next-generation carbon capture and storage (CCS) domains.

**Keywords:** corrosion, glass transition temperature, crosslinking, seawater, hydrocarbons, oil and gas, pipelines, batch reactor.



**Figure 1:** (a) Scanning electron microscopy (SEM) image showing corrosion products (later confirmed as oxides of iron by elemental mapping) deposited on the coating surface (marked with red circles) when exposed to HPHT conditions, and (b) the cross sectional SEM image of the exposed panel displaying corrosion products at the coating-steel interface (marked by red circles). The inset picture shows the area of SEM scan (marked by the black arrow). After [3].

## References:

1. Shadravan, A., & Amani, M. (2012, December). HPHT 101-what every engineer or geoscientist should know about high pressure high temperature wells. In SPE Kuwait International Petroleum Conference and Exhibition. OnePetro.
2. Rajagopalan, N., Weinell, C. E., Dam-Johansen, K., & Kiil, S. (2021). Progress in Organic Coatings, 156, 106268.
3. Rajagopalan, N., Weinell, C. E., Dam-Johansen, K., & Kiil, S. (2021). Influence of CO<sub>2</sub> at HPHT Conditions on the Properties and Failures of an Amine-Cured Epoxy Novolac Coating. Industrial & Engineering Chemistry Research, 60(41), 14768-14778.

# Mechanisms of corrosion protection of steel using industrial water-borne zinc rich sol-gel coatings

A. Boidot<sup>1,2,\*</sup>, C. Jama<sup>1</sup>, J-B. Vogt<sup>1</sup>, F. Gheno<sup>2</sup>

<sup>1</sup> Univ. Lille, CNRS, INRAE, ENSCL, UMR 8207 - UMET - Unité Matériaux et Transformations, F-59000 Lille, France

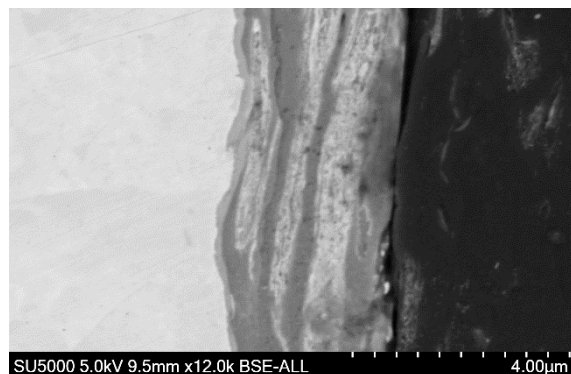
<sup>2</sup> NOF METAL COATINGS EUROPE, 120 rue Galilée, 60106 CREIL Cedex – France

## Abstract:

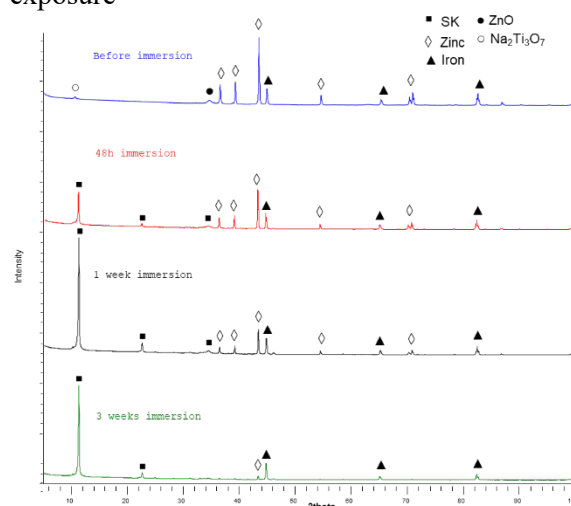
In this work, a sol-gel process is used to produce organic/inorganic hybrid zinc rich coatings for corrosion protection of steel. Compared to others, the sol-gel process has several advantages, it is often classified as an environmentally friendly technology since the processing temperature is generally low, chrome free and limits the emission of volatile organic compounds. On the other hand, zinc coatings are efficient options to overcome corrosion issues. Among them, zinc rich sol-gel coatings are a specific type made of zinc particles usually maintained together with an organic resin or a silicate binder, either solvent-based or waterborne and have been developed during the last decades. The evolution of this technology has made it possible to develop effective anticorrosion thin films, thus opening up a wider range of possible applications, particularly in the field of fasteners.

The study describes the mechanisms of corrosion protection of steel by the use of hybrid water-borne zinc flake sol-gel coatings (Figure 1) in a 30 g/L NaCl corrosive medium. Different formulas were tested before and after immersion in the aggressive medium for several weeks. Electrochemical Impedance Spectroscopy and Inductively Coupled Plasma Optical Emission Spectrometry techniques were used and gave a deeper understanding of the interfacial mechanisms and chemical reactions taking place in the system, showing that the film is actually increasing its barrier effect over time. Scanning Electron Microscopy and X-Ray Diffraction (Figure 2) were also performed and corroborated the previous results as they highlighted the formation of zinc corrosion products through the coating. Finally, some mechanical tests were also carried out and combined to corrosion tests to determine the ability of the coating to maintain protection despite mechanical loading.

**Keywords:** Corrosion protection, sol-gel coating, zinc-rich coating, corrosion products, electrochemical impedance spectroscopy



**Figure 1:** Backscattered electron image of the cross-section of a hybrid zinc rich coating before exposure



**Figure 2:** Diffractogram of the surface of a hybrid zinc rich coating, before and after different durations of exposure to 30 g/L NaCl

## References:

1. Wang, D., Bierwagen, G. (2009) Sol-gel coatings on metals for corrosion protection, *Prog. Org. Coatings.*, 64, 327–338.
2. Kalendova, A., (2003) Effects of particle sizes and shapes of zinc metal on the properties of anticorrosive coatings, *Prog. Org. Coatings.*, 46, 324–332.
3. Bierwagen, G., Allahar, K., Hinderliter, B., Jung, H., (2007) Zn-rich Coatings Revisited, *Tri-Service Corros. Conf.*



# Pull-off strength of polyurethane coatings with waste rubber fillers on aluminum substrate after aged and thermal shock processes

P. Mayer <sup>1\*</sup>, M. Ferraris <sup>2</sup>, S. Perero <sup>2</sup>

<sup>1</sup> Department of Lightweight Elements Engineering, Foundry and Automation, Wroclaw University of Science and Technology, Wroclaw, Poland

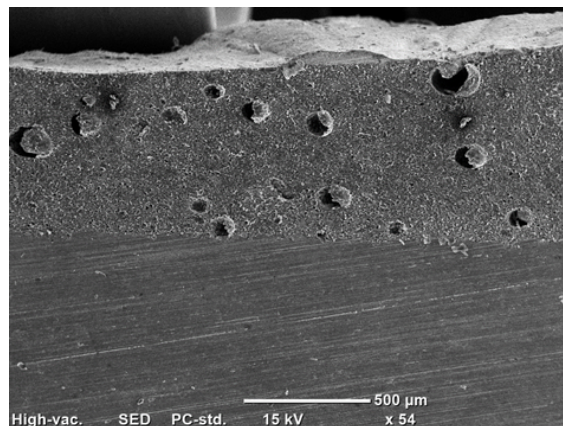
<sup>2</sup> Department of Applied Science and Technology, Politecnico di Torino, Torino, Italy

## Abstract:

The method of preparing the substrate is an important factor affecting the adhesion of the coatings. The aging processes that polymer materials are subjected to may adversely affect the adhesion of coatings to the substrate. The influence of surface roughness and aging process on adhesive joint strength for polymer coating reinforced with various rubber fillers to aluminium substrate was investigated. As fillers, SBR (styrene-butadiene) and EPDM (ethylene propylene diene monomer) rubber waste were used. The resin and accelerator were first mixed and 20% by weight of filler was added. PA11 aluminium alloy was chosen as the substrate, which was prepared in two different methods: using a primer or abrasive blasting. Two different abrasives (F80 alumina or GH40 cast steel shot) were used for mechanical treatment to obtain different roughness parameters. The coatings were made of polyurethane matrix and two different reinforcement materials EPDM and SBR rubber fillers. 100 cycles of thermal shocks were tested. One cycle is an hour, samples were fed at 100 degrees Celsius for half an hour and for another half an hour minus 40 degrees Celsius.

The pull-off strength test was performed using the electronic hydraulic coating adhesion meter PosiTest AT-A20 by DeFelsko. This device is adapted to perform the pull-off measurement described in the PN-EN ISO 4624: 2016 standard. Microscopic examination of the samples before the aging process was performed using an optical and electron microscope.

**Keywords:** polymer coatings, waste rubber fillers, aging process, shock thermal process, pull-off strength, gloss measurements, roughness measurements, polyurethane coatings, abrasive blasting, electron microscope



**Figure 1:** Figure illustrating the microscopic photo of a polyurethane coating with 20% by weight of EPDM powder filler. The coating is applied to an aluminum substrate that has been abrasive blasted with GH40 steel shot.

## References:

1. Guermazia, N., Elleuch K., Ayed, K.H., Kapsa, Ph., (2008) Aging effect on thermal, mechanical and tribological behaviour of polymeric coatings used for pipeline application, *J. Mater. Process. Technol.*, 203, 404-410.
2. Chowaniec, A., Sadowski, Ł., Żak A., (2020), The chemical and microstructural analysis of the adhesive properties of epoxy resin coatings modified using waste glass powder, *Appl. Surf. Sci.*, 504, 144373.

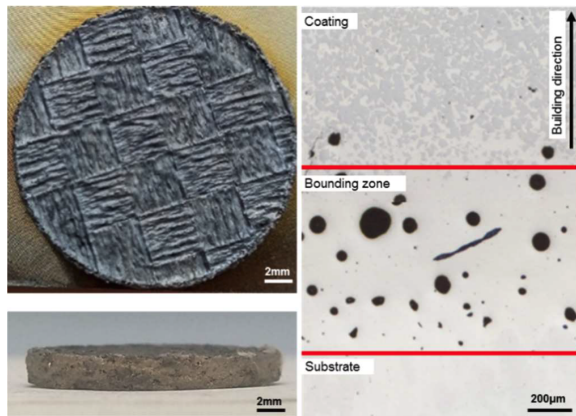
# Tungsten carbide surfaces: challenging PVD coatings by additive manufacturing

Florian Koehn<sup>1</sup>, Wadim Schulz<sup>1</sup>, Joachim Albrecht<sup>1</sup>

<sup>1</sup> Research Institute for Innovative Surfaces (FINO), Aalen University  
\*florian.koehn@hs-aalen.de

## Abstract:

The mechanical protection of component surfaces against wear is typically achieved by the deposition of hard coatings. Depending on the kind of application suitable material combinations and deposition techniques have to be selected. Co-based tungsten carbide coatings are generally obtained by physical vapor deposition (PVD)<sup>[1]</sup>. However, a promising approach uses 3D printing to produce such coatings with enhanced adhesion and extreme wear resistivity<sup>[2]</sup>. Comparing both coatings in tribological tests under severe wear inducing conditions, it can be found that the wear rate of additive manufactured coatings is more than one order of magnitude smaller as known for high quality PVD coated surfaces. However, the coefficient of friction highly depends on the surface topography<sup>[3,4]</sup>. It can also be obtained that microstructuring the surface leads to an improved wear behavior.



## Figure:

Figure 1 shows the additive manufactured WC/Co samples<sup>[2]</sup>. The left side depicts photographic images of a round sample printed in checkboard patterns to minimize thermal stress properties. On the right there are light microscope images of the cross section of the sample. It is separated into the three phases Substrate, Bounding zone and the actual WC/Co coating.

## Keywords:

tribology, coefficient of friction, microstructuring, additive manufacturing, SLM, PVD, tungsten carbide, wear resistivity

## References:

1. L. Haus, M. Wildfeuer, J.-E. Grochowski, J. Wöckel, M. Müller, F. Köhn, W. Schulz, C. Wüstefeld, D. Rafaja, J. Albrecht (2022) Wear properties of carbon-rich tungsten carbide films, *Wear*, 20146
2. F. Koehn, M. Sedlmajer, J. Albrecht, M. Merkel (2021) Additive Manufacturing of Tungsten Carbide Surfaces with Extreme Wear Resistivity, *Coatings*, 1240
3. T. Sube, M. Kommer, M. Fenker, B. Hader, J. Albrecht (2017) Reduced friction on  $\mu$ -Mo2N coatings deposited by high power impulse magnetron sputtering on microstructured surfaces, *Tribology International*, 106, 41-45
4. M. Kommer, T. Sube, A. Richter, M. Fenker, W. Schulz, B. Hader, J. Albrecht (2018) Enhanced wear resistance of molybdenum nitride coatings deposited by high power impulse magnetron sputtering by using micro-patterned surfaces, *Surface & Coatings Technology*, 333, 1-12



# Adhesive and wear behavior of a duplex coating of a nitrided layer plus diamond-like carbon coating.

I. Gómez<sup>1,2,\*</sup>, A. Claver<sup>1,2</sup>, J.A. Santiago<sup>3</sup>, J. Fernandez<sup>4</sup>, I. Bracerás<sup>5</sup>, J.A. Garcia<sup>1,2</sup>,

<sup>1</sup> Materials science, Public University of Navarre, Pamplona, Spain

<sup>2</sup> INAMAT (Institute for Research in Advanced Materials), Pamplona, Spain

<sup>3</sup> Nano4Energy SL, José Gutiérrez Abascal 2, 28006 Madrid, Spain

<sup>4</sup> Centre of Advanced Surface Engineering, AIN, 31191 Cordovilla, Spain

<sup>5</sup> Energy and Environment Division, Tecnalia Research & Innovation, Mikeletegi Pasealekua 2, 20009, Donostia - San Sebastián, Spain

## Abstract:

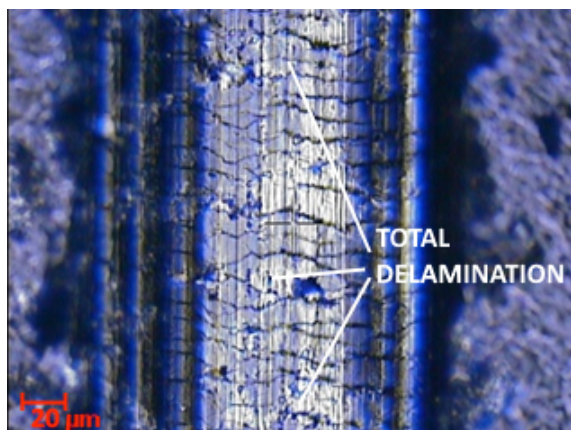
Diamond-like carbon (DLC) coatings are of great interest for properties such as wear resistance, low coefficient of friction, and chemical inertness. Because of this, they are used in tools and performance components in the automotive industry, improving the performance of sharp edges, biomedical components, dielectric barriers or barrier films on plastic.

For biomedical applications, diamond-like carbon coatings on austenitic 316L stainless steel have been studied to improve their properties. For this, duplex coatings composed of a nitrided layer plus a diamond-like carbon coating have been analyzed [1]. The DLC has been performed using the HiPIMS technique using positive pulses [2] and two nitriding layers have been proposed, plasma immersion ion implantation and active screen plasma nitriding.

Friction and wear tests were carried out by means of a balla on disc tribometer, hardness tests by nanoindentations as well as adhesion analysis by scratch test.

The results show us improvements in both adhesion (Figure 1) and wear resistance of said duplex coatings.

**Keywords:** HiPIMS, Positive pulses, DLC coatings, active screen plasma nitriding, Plasma immersion ion implantation, PIII, Adhesion, Wear resistance.



**Figure 1:** Figure illustrating the total delamination (LC3) of the ASPN duplex coating (400°C) + DLC after subjecting it to a scratch test whose delamination occurred at a load of 82N.

## References:

1. G. Iñigo, A. Santiago, J.F. Palacio, C. Diaz, S. Mändl, J.A. Garcia, Improved Adhesion of the DLC Coating Using HiPIMS with Positive Pulses and Plasma Immersion Pretreatment, (2021).
2. J.A. Santiago, I. Fernández-Martínez, A. Wennberg, J.M. Molina-Aldareguia, M. Castillo-Rodríguez, T.C. Rojas, J.C. Sánchez-López, M.U. González, J.M. García-Martín, H. Li, V. Bellido-González, M.A. Monclús, R. González-Arrabal, Adhesion enhancement of DLC hard coatings by HiPIMS metal ion etching pretreatment, *Surf. Coatings Technol.* 349 (2018) 787–796.

# Wear behavior of single stage HVOF sprayed TiNbMoMnFe high entropy alloys coating

N.V. Abhijith<sup>1\*</sup>, A. Pattanayak<sup>1</sup>, A. Gupta<sup>1</sup>, D. Kumar<sup>1</sup>

<sup>1</sup> Centre for Automotive Research and Tribology, Indian Institute of Technology Delhi, New Delhi, India

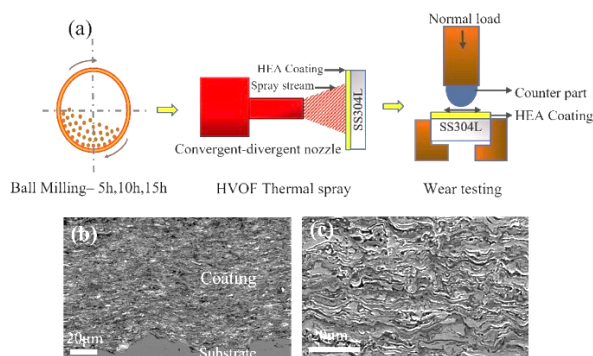
## Abstract:

Nowadays, a new group of alloys, namely high entropy alloys (HEA), is getting much attention as a new approach to surface engineering. However, the fabrication of HEAs requires multistage techniques, especially milling, sieving, compaction, sintering, inert media, etc. These processes are laborious, costly, time-oriented, and not suitable for commercial application. In this study, a single-stage process-based HVOF thermal spray was adopted to develop TiNbMoMnFe HEA coating on SS304L substrates. The wear behavior of the deposited HEA coating was explored under different milling time durations (5h, 10h and 15h, respectively). The effect of feedstock preparation, microstructure, hardness and phase distribution on wear resistance was also investigated. The microstructure and composition of both coating and feedstock were evaluated by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) analysis. Wear resistance was formulated in terms of coefficient of friction and wear loss. Finally, the phase distribution was correlated by XRD analysis. The results showed that 15h milled powder coating indicated better wear resistance than the base substrate and 5h, 10h milled powder coating. A chemically stable BCC solid solution phase was generated within the 15h milled powder-coated system, and which resulted in the higher wear resistance.

**Keywords:** High entropy alloys coating, wear mechanism, HVOF coating, microstructure

## References:

1. Löbel, M., Lindner, T., Mehner, T. and Lampke, T. (2017) Microstructure and Wear Resistance of AlCoCrFeNiTi High-Entropy Alloy Coatings Produced by HVOF, *Coatings*, 7(9), p. 144.
2. Meghwal, A., Anupam, A., Murty, B.S., Berndt, C.C., Kottada, R.S., and Ang, A. S. M. (2020), Thermal Spray High-Entropy Alloy Coatings: A Review, *J. Therm. Spray Technol.*, 2020, 29(5), p 857–893.
3. Chen, L., Bobzin, K., Zhou, Z., Zhao, L., Öte, M., Königstein, T., Tan, Z., He, D., (2019) Wear behavior of HVOF-sprayed Al<sub>0.6</sub>TiCrFeCoNi high entropy alloy coatings at different temperatures, *Surf. Coat. Tech.*, 358, p. 215–222



**Figure 1:** The process layout of (a) single stage HEA coating and (b,c) coating-substrate interface under different magnification

# Tribological Characterization of Ti-based Magnetron Sputtered Thin Films: A small-scale *in situ* study

A. Sayilan<sup>1,2</sup>, J. Ferreira<sup>1</sup>, C. Goudin<sup>1</sup>, C. Lopes<sup>3</sup>, J. Borges<sup>3</sup>, F. Vaz<sup>3</sup>, N. Mary<sup>1</sup>, S. Descartes<sup>2</sup>, P. Steyer<sup>1</sup>

<sup>1</sup>MATEIS, Univ. Lyon, INSA-Lyon, Villeurbanne, France

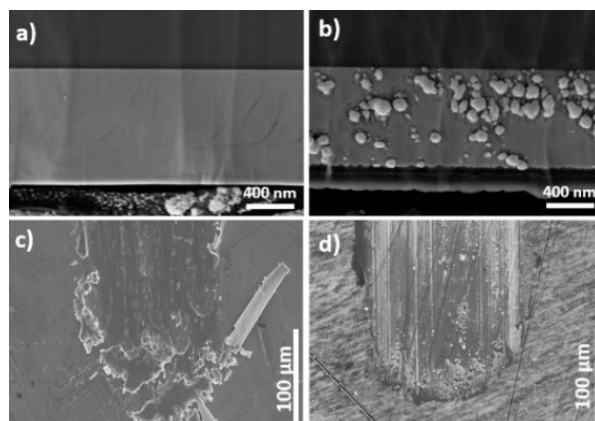
<sup>2</sup>LaMCoS, Univ. Lyon, INSA-Lyon, Villeurbanne, France

<sup>3</sup>Department of Physics, Univ. Minho, Braga, Portugal

## Abstract:

The nature of interface in contact with living species is the prime interest for bio-material applications. (biosensors, prosthesis, bio-implants...) Chemistry, structure and microstructure of this interface can be modified with physical vapour deposition process. Previously, for the biosensors, intense research activities were dedicated to Ti-Ag PVD thin films regarding the biocompatibility of titanium, and anti-bacterial character of silver. The electrical conductivity and the flexibility characters of these films have been studied<sup>1</sup>; but, their damaging mechanism against rubbing was not analysed before. Nevertheless, their wear characteristic is a significant issue in order to better understand the friction of bio-sensors against clothes, skin and human body. In this study, the objective is to examine the effect of film composition and environment on the tribological behaviour of Ti-Ag films in small scale *in situ* conditions. Ti-Ag films were deposited with magnetron sputtering PVD in various Ag/Ti (at.) ratios: pure Ti, low-, moderate- and high-Ag contents. The tribological characterization of these films were done with a novel approach which is a laboratory-made reciprocating ball-on-disc micro-tribometer. The device can be introduced into SEM chamber and also operable under Raman spectrometer and it provides *in situ* characterization of the wear track in small scale. The tribological tests with this device can be conducted under high and low vacuum and under different atmospheric conditions (humid, inert gas...) into environmental SEM chamber. First tests were performed in order to analyse the effect of film composition for Ti-Ag films. It was seen that Ag-richest composition (Ag/Ti(at.)=3.14) performed enhanced tribological character thanks to the solid lubricant effect of Ag-based clusters which were formed during deposition whereas there are spallation and lack of adhesion problems occurred for the other compositions. (Figure 1)

**Keywords:** small scale tribology, micro-tribometer, environmental-SEM, PVD, thin films, *in situ*, titanium, silver



**Figure 1:** Cross sections and wear tracks of a,c) Ti-rich, b,d) Ag-rich Ti-Ag films

## References:

1. Etiemble, A., Lopes, C., Bouala, G., Borges, J., Malchère, A., Langlois, C., Vaz, F., Steyer, P. (2019) Fracture resistance of Ti-Ag thin films deposited on polymeric substrates for biosignal acquisition applications, *Surf. Coat. Tech.*, 358, 646.

# Controlling the tribological properties of hard coatings by substrate engineering

W. Schulz<sup>1\*</sup>, F. Köhn<sup>1</sup>, J. Albrecht<sup>1</sup>

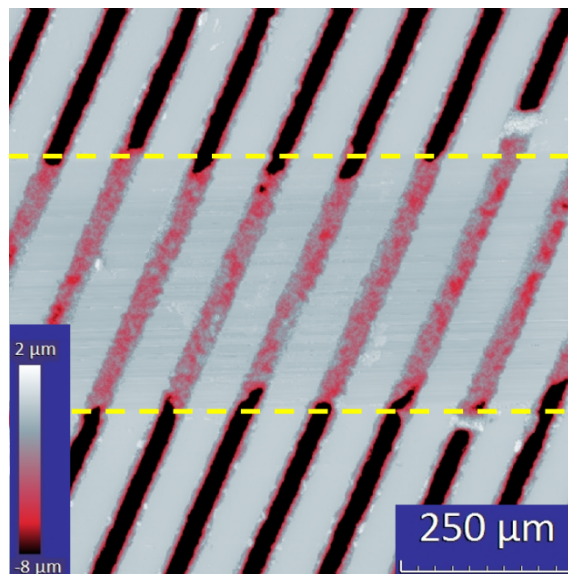
<sup>1</sup> Research institute for innovative surfaces (FINO), Aalen University, Aalen, Germany

## Abstract:

Friction and, in particular, wear at component surfaces regularly pose challenges for engineers. The key here is to find suitable material pairings or surface modifications to achieve the desired effects. A promising method is the microstructuring of affected surfaces. Methods such as laser surface texturing (LST) have already become established. It has been shown that the prior creation of appropriate structures can significantly reduce the contact areas of the surfaces moving relative to each other and thus the occurring wear [1]. In this context, the density of the structure elements plays a key role, anisotropic structures can further reduce the friction [2].

The abrasive particles produced in the tribological process cause enormous damage to the respective surfaces. The combination of surface structure and hard coating allows such particles to be trapped (Figure 1) and removed from the frictional contact, which in turn results in an increase in service life [3]. In the case of linear structures, an angular dependence of the structure on friction and wear is evident. Due to the variation of the structure angle in relation to the direction of stress, the wear on the layer and the counterbody is different [4].

**Keywords:** friction, wear, surface engineering, laser surface texturing (LST), surface technology, tribology



**Figure 1:** Textured and hard-coated surface after a tribological load. The yellow lines mark the wear track. Shown in red are the trapped particles of the counterbody from the frictional contact.

## References:

1. Zhu, Y., Chen, J., Du, J., Fan, Y., Zheng, J. (2019), Tribological behaviour of laser textured nodular cast iron surface, *Int. Lubr. Tribol.*, 71/7, 949-955.
2. Rosenkranz, A., Reinert, L., Gachot, C., Mücklich, F. (2014), Alignment and wear debris effects between laser-patterned steel surfaces under dry sliding conditions, *Wear*, 318, 49-61.
3. Kommer, M., Sube, T., Richter, A., Fenker, M., Schulz, W., Hader, B., Albrecht, J. (2018), Enhanced wear resistance of molybdenum nitride coatings deposited by high power impulse magnetron sputtering by using micropatterned surfaces, *Surf. Coat. Technol.*, 333, 1-12.
4. Schulz, W., Köhn, F., Kolb, D., Balzer, M., Riegel, H., Albrecht, J. (2021), Controlling Friction and Wear with Anisotropic Microstructures in MoN-Coated Surfaces, *Tribol. Lett.*, 69, 152.



# Effect of Substrate (WC-Co) Surface Roughness on the Tribological Properties of DLC Coatings Deposited by HiPIMS

Sharjeel Ahmed Khan <sup>1,2,\*</sup>, João Oliveira <sup>1</sup>, Nazanin Emami <sup>2</sup>, Amilcar Ramalho <sup>1</sup>

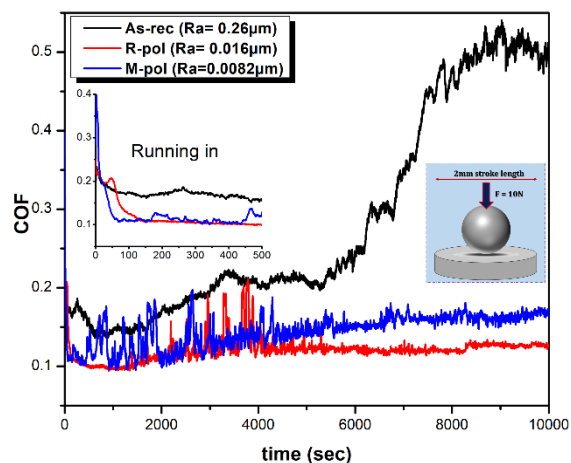
<sup>1</sup> Department of Mechanical Engineering, CEMMPRE, University of Coimbra, Coimbra, Portugal

<sup>2</sup> Department of Engineering Sciences and Mathematics, Luleå University of Technology, Luleå, Sweden

## Abstract:

The application of DLC coatings in dry machining of difficult-to-machine components have been gaining popularity due to the high inertness, low coefficient of friction and high hardness. The effect of surface roughness on the tribological properties of coatings is of paramount importance but often overlooked; as coatings performance analysis were mostly accomplished on highly polished surfaces. The creation of mirror polished surfaces is labour-intensive, and in most circumstances not feasible for the industry for being a high energy and time consuming process<sup>[1]</sup>. Beside that, a minute grinding operation could reduce peak point of surface roughness and significantly improve the load bearing capabilities and tribological properties of the coatings. This work focuses on determining the effect of substrate (WC-Co) surface roughness on the load bearing capacity and tribological properties of the DLC coatings deposited by High Power Impulse Magnetron Sputtering (HiPIMS) in Ne gas plasma<sup>[2]</sup>. In this study, three different substrate with varying surface roughness (as-recieved surface (As-rec), Roughly polished (R-pol) and Mirror polished (M-pol)) were analysed through scratch testing and their tribological performance were tested in a reciporocating tribotest against 100Cr6 steel ball. The high surface roughness of substrate resulted in increased COF and wear due to the high level of asperities and can results in premature failure of the coatings from the substrate meanwhile causing swear wear to the counter body.

**Keywords:** WC-Co, HiPIMS, DLC coatings, surface roughness, Wear, Adhesion



**Figure 1:** Variation of COF of DLC coatings, deposited on WC-Co substrate of different surface roughness using HiPIMS technique. The inset graph illustrate the running-in for a time upto 500 sec. For As-rec sample, the COF doesnot reached a steady state instead increases with time in comparison with other samples (R-pol and M-pol).

## References:

1. Vican M, Bokůvka O, Nikolić RR, Bronček J. Influence of the surface roughness of the C55 steel on its tribological properties after application of the WC / C coating. Transp Res Procedia 2021;55:490–5.
2. Ferreira F, Aijaz A, Kubart T, Cavaleiro A, Oliveira J. Hard and dense diamond like carbon coatings deposited by deep oscillations magnetron sputtering. Surf Coatings Technol 2018;336:92–8.

# Antiadhesive coating in aqueous phase to prevent colonization by microorganisms

M. Champion <sup>1\*</sup>, I. Linossier <sup>1</sup>, K. Rehel <sup>1</sup>, C. Hellio <sup>2</sup>, X. Moppert <sup>3</sup>, F. Fay <sup>1</sup>

<sup>1</sup> Laboratoire de biotechnologie et chimie marines, Université de Bretagne sud, Lorient, France

<sup>2</sup> Institut universitaire européen de la mer, Université de Bretagne occidentale, Plouzané, France

<sup>3</sup> Pacific Biotech, Arue, Polynésie française

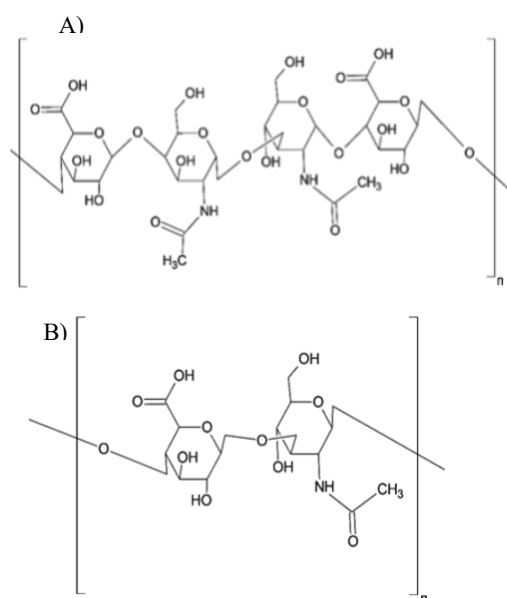
## Abstract:

Submerged surfaces are impacted by microorganisms' colonization causing many health and environmental concerns. To prevent this colonization, coatings are applied to the surfaces. These coatings often contain biocidal agents, leading to problems of pollution and resistance[1]. Current strategies tend to develop passive surfaces, free of biocides, limiting colonization solely through their physical, mechanical and chemical properties.

The marine environment is full of many polymers with interesting film-forming properties. In this work two types of polymers will be studied. Firstly, chitosan, a biosourced polymer with modulable mechanical properties, is a good candidate for the design of coatings. Chitosan is already known for the design of hydrophilic cross-linked networks[2]. The ability of chitosan to be cross-linked will allow the mechanical properties and thus us to establish different formulations, thus varying the cross-linking. The second polymers studied are exopolysaccharides (EPS). They are hydrophilic polymers produced by bacteria and having interesting anti-adhesive properties. It is an EPS produced by a marine bacterium from French Polynesia of the genus *Vibrio*. This EPS is described as having structural properties similar to hyaluronic acid (HA) (figure1), also susceptible to make cross-linked hydrogels.

The objective of this study is to design different types of hydrophilic coatings in order to mask the surfaces to the organisms thanks to a hydration layer. The modulation of the physical, mechanical and chemical properties of the coatings will allow us to understand the impact of hydrophilic coatings on the adhesion of organisms (bacteria, diatoms and algae spores) and thus to propose new alternatives to coatings containing biocides.

**Keywords:** antiadhesive coating, exopolysaccharide, polymers, biomaterials, surface hydration, , biomedical applications, marine applications, hydrophilic coating.



**Figure 1:** Figure illustrating MO245 and HA structure. A) EPS hyaluronic acid like, B) HA

## References:

1. Jiang P, Li J, Han F, et al. (2011) Antibio-film Activity of an Exopolysaccharide from Marine Bacterium *Vibrio* sp. QY101. *PLoS ONE* 6: e18514.
2. Ravishankar K, Dhamodharan R (2020) Advances in chitosan-based hydrogels: Evolution from covalently crosslinked systems to ionotropically crosslinked superabsorbents. *Reactive and Functional Polymers* 149: 104517.



## **SICT 2022 / Tribology 2022 joint Session II:**

### **Physics or chemistry of tribo- surfaces/ Nanotribology (Part I)**

# Adhesion-aided friction of viscoelastic materials

G. Carbone<sup>1,2,\*</sup>, C. Mandriota<sup>1</sup>, N. Menga<sup>1</sup>

<sup>1</sup> Department of Mechanics, Mathematics and Management, Polytechnic University of Bari, Bari, Italy

<sup>2</sup> CNR Institute for Photonics and Nanotechnologies U.O.S. Bari, Physics Department M. Merlin, Bari, Italy

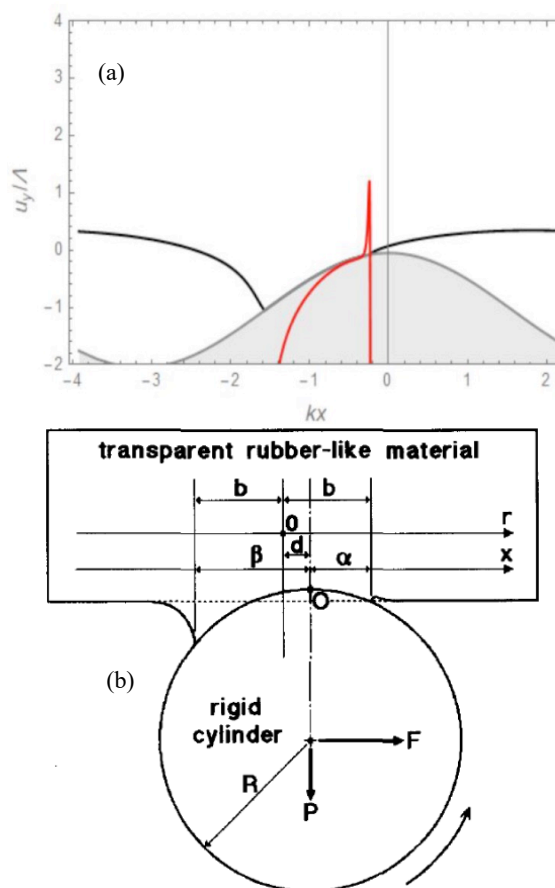
## Abstract:

In this lecture, we present preliminary results obtained by our recent developed theory of adhesive viscoelastic contacts [1]. The theory explains most of the experimentally observed phenomena emerging from the combined effect of viscoelasticity and adhesion. The study covers both local viscoelasticity, occurring at the tip of propagating adhesive cracks and bulk viscoelasticity, over velocities ranging from extremely low to extremely high values.

We explain why a bell shaped behaviour of the energy release rate is observed experimentally in finite systems, and predict the experimental observation by Charmet and Barquins [2], and those by Roberts [3]. The latter, in particular, shows that the viscoelastic friction is strongly enhanced by adhesion, for velocities below a threshold value, beyond which visco-adhesive friction vanishes and bulk-viscoelasticity becomes dominant.

Our theory provides fundamental insights into the physical mechanisms that govern interaction phenomena occurring upon contact between two surfaces, thus it plays a major role in a large number of physical phenomena and engineering applications as in the case of structural adhesives, pressure sensitive adhesives (PSA), protective coatings, tire friction, windscreen wipers, lubrication, wear, sealings, to mention some examples.

**Keywords:** visco-adhesive friction, adhesion, contact mechanics, viscoelasticity, polymers, soft contacts, tribology.



**Figure 1:** Figure illustrating the asymmetric contact originated by the combined effect of viscoelasticity and van der Waals adhesive forces at the interfaces, in a sliding/rolling contact. Results: (a) theory, (b) experiments (figure adapted from Ref. [2])

## References:

1. Carbone G., C. Mandriota, N. Menga, Theory of viscoadhesive contacts, *Physical Review Letters*, in preparation, (2022).
2. J.-C. Charmet and M. Barquins, Adhesive contact and rolling of a rigid cylinder under the pull of gravity on the underside of a smooth-surfaced sheet of rubber, *International Journal of Adhesion and Adhesives*, **16**, 249-254, 1996.
3. A. D. Roberts, Looking at Rubber Adhesion, *Rubber Chemistry and Technology* **52** (1), 23-42, (1979)

# Compositional and topographical tailoring of tungsten carbide surfaces to control friction and wear under dry conditions

J. Albrecht, W. Schulz, F. Köhn

Research Institute for Innovative Surfaces FINO, Aalen University, Beethovenstr. 1, D-73430, Aalen, Germany

## Abstract:

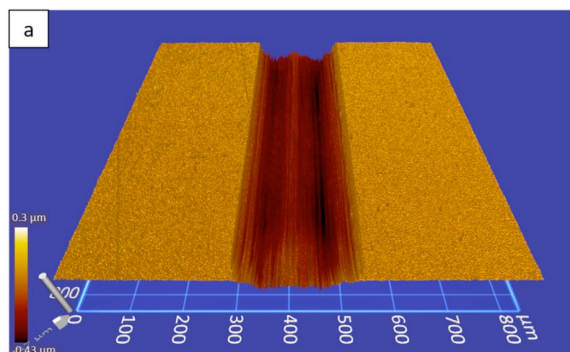
Modifications of the surface topography of hard coatings on the microscale can lead to a substantial reduction of friction and wear [1,2]. Alternatively, beneficial modifications of the mechanical properties can be obtained by compositional variations of the wear-resistant material introduced by a systematically modified deposition process. In this work results are presented that are obtained on thin films of tungsten carbide that are prepared by physical vapor deposition. The films were deposited on steel substrates by magnetron sputtering of WC in a mixed argon/acetylene atmosphere. Variation of the acetylene supply during the deposition led to the growth of tungsten carbide films with different carbon excess. Variation of the bias voltage affects both the stoichiometry and the mechanical properties. The films are supposed to combine good wear-resistant properties with a low friction under dry conditions. A tribometric analysis of friction and wear under heavy load and dry conditions shows that an excess of carbon can reduce the coefficient of friction by up to 40%. However, an increase of the wear volume is found with higher carbon content [3]. A comparison to additively manufactured, bulky tungsten carbide surfaces [4] shows that an optimization of wear-resistive tungsten carbide surfaces can be found when considering the microstructure, the composition and finally also the properties of the substrate underneath.

**Keywords:** Hard coatings, tungsten carbide, film deposition, friction, wear

testing against a 100Cr6 steel ball under high load for  $t = 10$  min [3].

## References:

1. T. Sube, M. Kommer, M. Fenker, B. Hader, and J. Albrecht, Reduced friction on  $\gamma$ -Mo<sub>2</sub>N coatings deposited by high power impulse magnetron sputtering on microstructured surfaces, *Trib. Int.* 106 (2017) 41–45.
2. M. Kommer, T. Sube, A. Richter, M. Fenker, W. Schulz, B. Hader and J. Albrecht, Enhanced wear resistance of molybdenum nitride coatings deposited by high power impulse magnetron sputtering by using micropatterned surfaces, *Surf. Coat. Technol.* 333 (2018) 1–12.
3. L. Haus, M. Wildfeuer, J.-E. Grochowski, J. Wöckel, M. Müller, F. Köhn, W. Schulz, C. Wüstefeld, D. Rafaja and J. Albrecht, Wear properties of carbon-rich tungsten carbide films, *Wear* 488–489 (2022) 204146.
4. F. Köhn, M. Sedlmajer, J. Albrecht and M. Merkel, Additive manufacturing of tungsten carbide surfaces with extreme wear resistivity, *Coatings* 11 (2021) 1240.



**Figure 1:** Topography of a wear track on a WC film with stoichiometric composition after dry

# Experimental and modelling based upscaling of new material concept for journal bearings

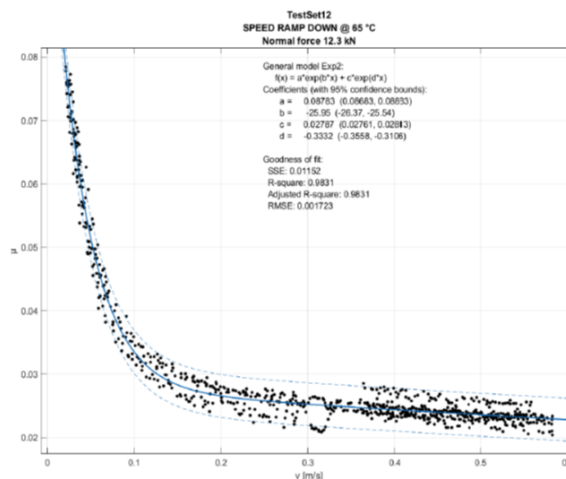
H. Ronkainen\*, J. Tervo, M. Savolainen, J. Virtanen, Sami Majaniemi

VTT Technical Research Centre of Finland, Espoo, Finland

## Abstract:

Journal bearing are used in a variety of applications to support rotating machinery. They are used widely in e.g. combustion engines and steam turbines [1], and lately they have been introduced also to gear units of wind turbines. In journal bearings the lubricant film is carrying the load, avoiding metal-metal contacts of the surfaces, and influencing the friction performance of the bearing. Therefore the lubrication status is essential for the proper performance of the journal bearings. The general challenge related to experimental studies of bearing performance is relates to interpretation of the results of small-scale tests to larger scale components. This presentation describes the laboratory scale journal bearing tests carried out and combined with MBS-FEM/EHD-modelling to provide data that can be used in the larger, pilot or product scale modelling as input values and for improving bearing performance and making estimates on e.g. the life-time of new material concepts for journal bearings.

The experimental laboratory scale journal bearing tests were carried out with a new material concept for journal bearings. The tests were carried out with different speeds ranging from 0.012 to 0.5 m/s and specific loads in the range 0.5 to 25 MPa. One target was to study the influence of operational parameters on the predominant lubrication regime and thus the friction performance of the new journal bearing material concept. Figure 1 describes as an example the evolution of friction coefficient in the speed ramp down -tests. The friction shows typical Stiebeck curve as the sliding speed was ramped from 0.5 m/s to 0.012 m/s and back to 0.5 m/s. The friction evolution describes the shift from fluid film lubrication to mixed lubrication and further to boundary lubrication. By utilizing the MBS-FEM/EHD modelling it is possible to study the range of sliding speed when the shift between the lubrication regimes occur. This information can be combined with the larger e.g. product scale models. Also long duration tests were carried out to study the wear performance of the bearing material concept and provide information for the life-time estimations.



**Figure 1:** The friction evolution as a function of sliding speed in the range 0.03 m/s to 0.5 m/. The specific load was 20.5 MPa and the temperature  $62 \pm 2^\circ\text{C}$  during the tests Test was repeated three times.

Based on experimental and modelling approach a modelling-based upscaling of experimental results can be carried out to enable the scale jump to bridge the gap between the laboratory test scale and the larger component scale. This approach can decrease the need to build expensive components for testing purposes and diminishes the number of higher scale experiments thereby speeding up the materials up-scaling process.

**Keywords:** journal bearing, friction, experimental, modelling, materials.

## References:

1. Khonsari M.M., and Booser E.R., Applied Tribology: Bearing Design and Lubrication. John Wiley & Sons Ltd, 2017.

## Acknowledgement

The project i-TRIBOMAT is financially supported by the European Union's Horizon 2020 research and innovation programme under Grant Agreement no. 814494 (for further information check: [www.i-tribomat.eu](http://www.i-tribomat.eu)).



# Challenges to Testing - Rolling Contact Fatigue on Modern High Strength Steels

V. Heino<sup>1\*</sup>, R. Parikka<sup>1</sup>, M. Lindroos<sup>1</sup>, H. Ronkainen<sup>1</sup>

<sup>1</sup> VTT Technical Research Centre of Finland Ltd, Espoo, Finland

## Abstract:

High quality, high strength steels are commonly used in applications where the rolling contact fatigue is one of the key failure modes, such as in gear contacts in wind-turbine gear box. The size of the wind turbines designed and build is increasing and the demand to ever increasing power to weight ratios is putting more pressure towards improved fatigue life of materials.

The larger the component size, the higher is the propability for the fatigue failure to occur, since statistically the occurrence of material impurities are higher than in smaller size components. This lead to increased down-time for the power plant or in worst case scenario (if sudden failure, without clear indications via machine diagnostics) total failure of whole gear box.

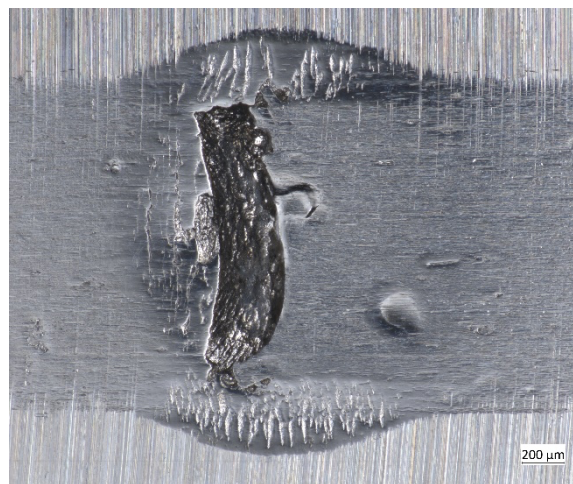
The key player related to the rolling contact fatigue is the purity of the material and also sufficient hardness of the surface region with moderate hardness gradient towards the inner section of the component. Therefore many of components that are in high risk to suffer the rolling contact fatigue failure, have high hardness surface and ductile inner section provided for example by case hardening.

The high quality and purity of these steels inevitably affect also the rolling contact testing. For instance with twin disc type of testing devices, the testing will take many weeks to show any signs of macro-pitting even with the higher loads. There is need to define the tests concept and pay more attention on the characterization of the subsurface region of the tested components.

This study relates to the defining the rolling contact features with accelerated rolling contact fatigue testing for case-hardened high strength steels. It was observed that by accelerating the rolling contact failures with artificial flaws which concentrate high contact pressures in the surface regions, rolling contact fatigue results can be obtained in reasonable time. However, it should be noted that with accelerated testing more precise analysis of the rolling contact failure mechanisms and the characterization of the material properties should be made. Combined with characterization, the crystal plasticity material model is

utilized in this study to increase understanding of the phenomena related to the fatigue behavior of these investigated high strength steel microstructures.

**Keywords:** rolling-contact fatigue, RCF, twin-disc, high-strength steels.



**Figure 1:** Macro-pitting failure on the disc during accelerated RCF testing.

## Acknowledgements:

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 851245

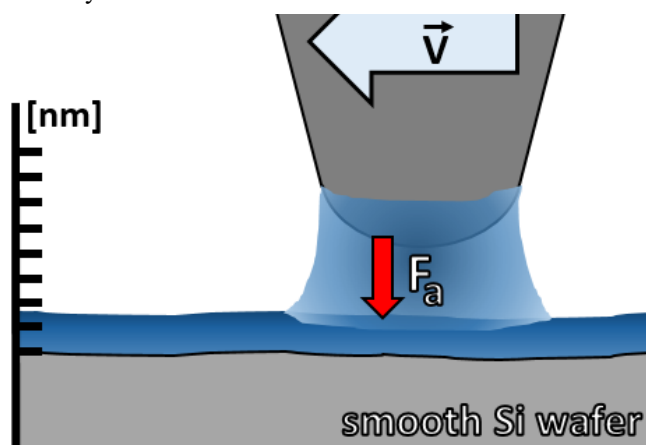


# The Dynamics of Capillary Bridge Formation in the Non-Contact Regime

F. Cassin<sup>1</sup>, B. Weber<sup>1</sup>

<sup>1</sup>Contact Dynamics, Advanced Research Center for Nanolithography, Amsterdam, The Netherlands

**Figure 1:** Experiment design where the AFM probe is kept at a stable distance above the smooth Si wafer with its native water layer.



The formation of liquid bridges between two sliding surfaces due to capillary condensation and its influence on friction and wear has become a critical field of research in nanotribology. Not only solid on solid contact allows for the formation of water bridges but also surrounding areas with a surface-to-surface distance of a few nanometers, close enough to form capillary bridges, can be much bigger and therefore contribute significantly to the normal load and friction of a sliding interface.

This study utilizes the Atomic Force Microscope (AFM) to understand the formation of capillary bridges between a 30nm radius probe and a laterally moving smooth Si wafer surface at different tip-sample distances

which is kept stable by with the help of the Lift Mode. We further investigate how the sliding velocity (0.2 to 100  $\mu\text{m/s}$ ) influences the nucleation rate and adhesion forces of the menisci formed at separation distances between 0 and 4 nm.

## References

1. Noel, O., Mazeran, P. E., & Nasrallah, H. (2012). Sliding velocity dependence of adhesion in a nanometer-sized contact. *Physical Review Letters*, 108(1), 1–4. <https://doi.org/10.1103/PhysRevLett.108.015503>
2. Sung, B., Kim, J., Stambaugh, C., Chang, S. J., & Jhe, W. (2013). Direct measurement of activation time and nucleation rate in capillary-condensed water nanomeniscus. *Applied Physics Letters*, 103(21). <https://doi.org/10.1063/1.4832879>



## Recent *in-silico* models for wear calculation in total hip replacements

A. Ruggiero

Department of Industrial Engineering, University of Salerno, Fisciano (Salerno), Italy

### Abstract:

Nowadays the biotribology of the human articulations is a challenging topic in the framework of the biomechanics research. A human synovial joint is a complex tribological system able to minimize friction and wear of the articulated cartilage surfaces, thanks to the mix of particular natural lubrication regimes which are established within the synovial membrane.

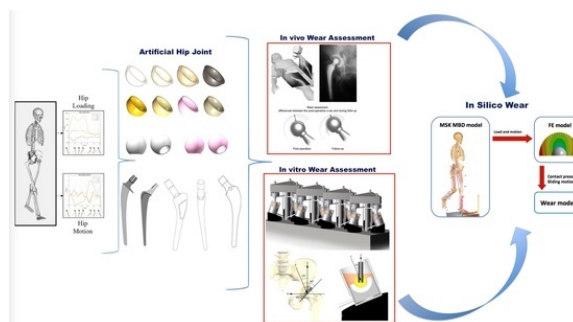
In the last years, the number of artificial hip joints, as well as the amount of necessary economic resources, are constantly growing [1]. This surgical procedure, thanks to the excellent clinical results obtained in the last decades, has evolved rapidly to solve degenerative diseases on the hip synovial joints. Hip arthroplasty is recognized as one of the most successful orthopedic surgical procedures, even if it involves challenges to overcome, such that lately, younger and more active patients are in need of total arthroplasty. After total or partial joint replacements, the articular functions are fully restored and the patients return to a pain-free condition [2]. In this framework it becomes necessary to guarantee that new prosthetic designs, in terms of geometry and adopted materials, have to be deeply analyzed and tested. Wear is still one of the main issues affecting joint prostheses endurance, and often causes loosening accompanied by implant failures. An accurate preclinical validation of these medical devices is necessary in order to establish their tribological performances and wear resistance.

Actual *in vitro* wear tests were executed by mechanical simulators and they have a long duration, are very expensive, and do not take into account all the possible daily activities of the patients. The challenge to obtain a complete *in-silico* tribological and dynamical model of (bio) tribo-systems could give the possibility to overcome the actual testing procedures and could contribute as a tool for a more accurate tribological design of human prostheses [3,4,5,6]. This speech is intended to underline actual research trends toward the challenge of having accurate numerical algorithms to be used both in preclinical testing and in the optimizations of the prostheses design. With this aim was depicted the possible *in-silico* approach in artificial joints' wear assessment over time, accounting for

contact mechanics, numerical stress–strain analysis, musculoskeletal multibody, and synovial lubrication modelling (boundary/mixed, hydrodynamic, and elastohydrodynamic).

**Keywords:** biotribology, THR, TKR, *in-silico* musculoskeletal multibody systems.

**Figure 1:** Toward the accurate Total Hip Replacements *in-silico* wear testing.



### References:

1. Affatato, S.; Ruggiero, A. A Perspective on Biotribology in Arthroplasty: From In Vitro toward the Accurate In Silico Wear Prediction. *Appl. Sci.* 2020, 10, 6312. <https://doi.org/10.3390/app10186312>.
2. Viceconti, M.; Affatato, S.; Baleani, M.; Bordini, B.; Cristofolini, L.; Taddei, F. Pre-clinical validation of joint prostheses: A systematic approach. *J. Mech. Behav. Biomed. Mater.* 2009, 2, 120–127.
3. Ruggiero, A., & Sicilia, A. (2020). Lubrication modeling and wear calculation in artificial hip joint during the gait. *Tribology International*, 142, 105993.
4. Ruggiero, A., Sicilia, A., & Affatato, S. (2020). In silico total hip replacement wear testing in the framework of ISO 14242-3 accounting for mixed elasto-hydrodynamic lubrication effects. *Wear*, 460, 203420.
5. Ruggiero, A., & Sicilia, A. (2020). A mixed elasto-hydrodynamic lubrication model for wear calculation in artificial hip joints. *Lubricants*, 8(7), 72.
6. Ruggiero, A., & Sicilia, A. (2021). Mathematical Development of a Novel Discrete Hip Deformation Algorithm for the In Silico Elasto-Hydrodynamic Lubrication Modelling of Total Hip Replacements. *Lubricants*, 9(4), 41.

# Wear Particle Dynamics Drives the Difference between Repeated and Non-Repeated Reciprocating Sliding

Feng-Chun Hsia<sup>1</sup>, Fiona Elam<sup>1</sup>, Daniel Bonn<sup>2</sup>, Bart Weber<sup>1,2</sup>, Steve Franklin<sup>1,3\*</sup>

<sup>1</sup> Advanced Research Center for Nanolithography (ARCNL), Amsterdam, The Netherlands

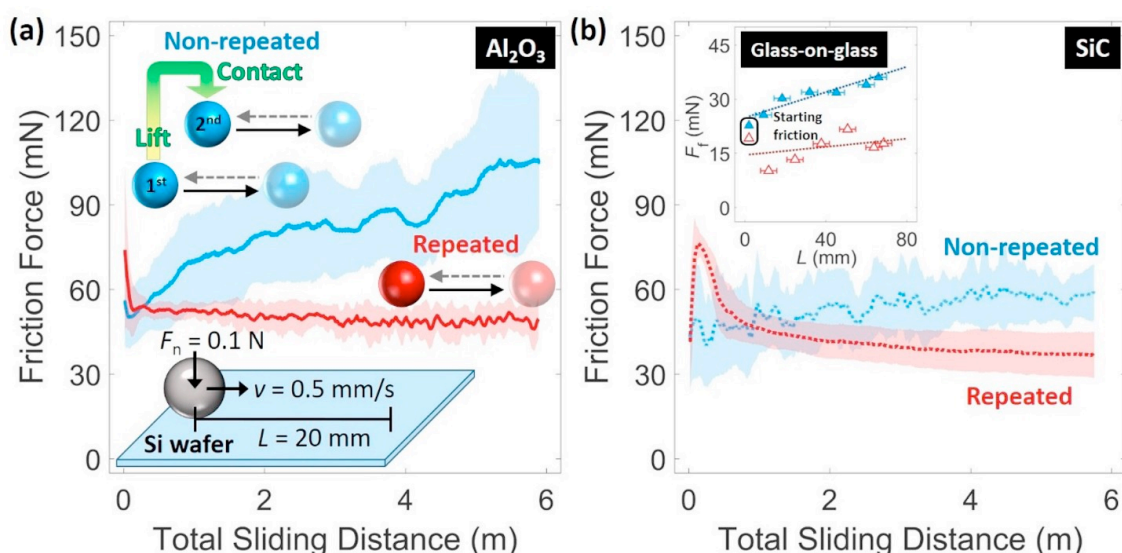
<sup>2</sup> Van der Waals-Zeeman Institute, IoP, University of Amsterdam, Amsterdam, The Netherlands

<sup>3</sup> Department of Materials Science & Engineering, The University of Sheffield, Sheffield, UK

## Abstract:

Although many applications involve repeated, reciprocated sliding of the same two surfaces against each other, in many other real applications sliding occurs in a non-repeated fashion, one surface always encountering a fresh, unworn counter-surface. Examples of the latter include the tooling in materials processing applications such as deep drawing, stamping and cutting, the read/write head of computer hard drives, and the tip used in atomic force microscopy imaging, especially when used in lateral force microscopy mode. However, experiments performed to reproduce the friction behaviour in applications are commonly carried out in a repeated fashion, for example using a pin-on-disk or repeated reciprocating test geometry, even when the actual application clearly involves non-repeated sliding. The potential consequences this has for the tribological behavior are often overlooked. Here, the dependence of the friction and wear behaviour on the sliding mode (repeated vs. non-repeated reciprocated sliding) is evaluated. Experiments were carried out with  $\text{Al}_2\text{O}_3$  (sapphire), silicon carbide (SiC) and glass balls sliding against silicon wafers or glass flats, in ambient air at 21–23 °C and 20–60% relative humidity (Figure 1). For the material systems and conditions tested, repeated sliding was found to promote the formation of a third body (compressed wear particles) that led to stabilisation of the friction. Non-repeated sliding showed much less evidence of third body formation, and instead a steady increase in friction. The proposed mechanism driving the non-repeated friction behaviour was attributed to a gradual reduction in the ball surface roughness, leading to an increased area of real contact and potentially greater capillary bridge forming and increased van der Waals forces across non-contact regions of the interface.

**Keywords:** friction, wear, adhesion, third-body.



**Figure 1:** Friction measurements during repeated and non-repeated sliding of (a) a sapphire ball on a Si wafer and (b) a SiC ball on a Si wafer. Insets in (a) show schematic illustrations of the sliding mode. The inset in (b) shows glass ball on glass flat friction measurements. The shaded areas indicate the standard deviation in the measured friction forces for a minimum of three identical independent experiments. For the glass-on-glass friction experiments (inset 1b) each data point represents the average friction force for 4 cycles.

# Fine Defect Engineering of Graphene Friction

A. Zambudio<sup>1,2</sup>, E. Gnecco<sup>3</sup>, J. Colchero<sup>1</sup>, R. Pérez<sup>4,5</sup>, J. Gómez-Herrero<sup>2,5</sup> and C. Gómez-Navarro<sup>2,5</sup>

<sup>1</sup>Optics and Nanophysics Research Center, Universidad de Murcia, 30100, Murcia, Spain

<sup>2</sup>Condensed Matter Physics Dept., Universidad Autónoma de Madrid, Madrid, E-28049, Spain

<sup>3</sup>Otto Schott Institute of Materials Research, Friedrich Schiller University of Jena, D-07743, Jena, Germany

<sup>4</sup>Theory Condensed Matter Physics Dept., Universidad Autónoma de Madrid, Madrid, E-28049, Spain

<sup>5</sup>Condensed Matter Physics Center, Universidad Autónoma de Madrid, Madrid, E-28049, Spain

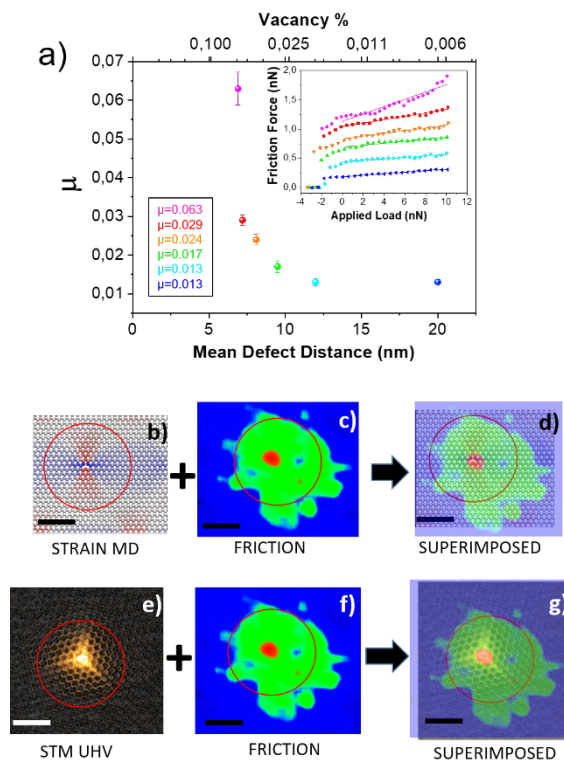
## Abstract:

Two-dimensional materials, in particular graphene, exhibits a low friction coefficient and good wear properties. However, the presence of atomic defects, inherent to any large-scale production, hugely influence the friction and other mechanical properties of graphene. Up to date, tribological studies of defective graphene combine coexistent substitution-like and vacancy defects with several defect size[1]. This leads to a difficult interpretation and comparison of results, and prevents from a fundamental understanding of the role of defects on graphene friction.

In this work, we quantify the influence of controlled-induced monoatomic vacancies in graphene tribology using Atomic Force Microscopy (AFM). This simplest and very common type of defects is demonstrated to increase the friction of graphene. Furthermore, friction coefficient is as well enhanced by defects in a highly efficient manner. Only 0.1% of defects yields to a five-fold increase of friction coefficient[2]. At the atomic-scale, we resolve monoatomic vacancies in friction images showing a great correlation with Prandtl-Tomlinson model atomic simulations.

Thorough analysis of real space distribution of friction data at the nanoscale reveals two main contributions to friction enhancement. One is related to reactivity of dangling bonds localized at monovacancy site ( $\sim 1\text{nm}^2$ ) and is responsible of  $\sim 20\%$  of the increase. The other is a more extended one ( $\sim 25\text{nm}^2$ ) and arises from the long-range strain distribution around vacancies, being the main contribution to friction enhancement on defective graphene. These results elucidate the subtle connection between friction, reactivity, and mechanical properties in two-dimensional materials..

**Keywords:** Graphene, friction, vacancy, tribology, defects



**Figure 1:** a) Friction coefficient at different mean defect distances, obtained from friction vs applied normal force curves depicted at upper inset. Lower inset shows extracted friction coefficient values. b) 2D stress map obtained from Molecular Dynamic simulations[3] in a region surrounding a carbon monovacancy. c) and f) Lateral force images centred on a carbon monovacancy. d) Superposition of panels b) and c). e) Experimental STM image of a carbon monovacancy acquired in UHV conditions. (courtesy of I. Brihuega) g) Superposition of panels e) and f). Both d) and g) panels show a clear spatial correlation between the defect influence area on friction images acquired in the present work and previous theoretical and experimental results on similar graphene monovacancies. Scale bar for b) to g) images is 2 nm..

## References:

1. Kwon, S., Ko, J.-H., Jeon, K.-J., Kim, Y.-H., Park, J. Y. (2012) Enhanced nanoscale

- friction of fluorinated graphene, *Nano-Letters*, 12, 6043-6048.
2. Zambudio, A., Gnecco, E., Colchero, J., Pérez, R., Gómez-Herrero, J., Gómez-Navarro, C. (2021) Fine defect engineering of graphene friction, *Carbon*, 182, 735-741.
  3. López-Polín, G., Ortega, M., Vilhena, G., Alda, I., Gómez-Herrero, J., Serena, P. A., Gómez-Navarro, C., Pérez, R. (2017) Tailoring the thermal expansion of graphene via controlled defect creation, *Carbon*, 116, 670-677.

# Investigation of experimental production parameters effects on the wear behaviour of copper-tungsten disulfide composite

M. Freschi<sup>1\*</sup>, L. Dragoni<sup>1</sup>, M. Mariani<sup>2</sup>, O. Haiko<sup>3</sup>, J. Kömi<sup>3</sup>, N. Lecis<sup>2</sup>, G. Dotelli<sup>1</sup>

<sup>1</sup> Department of Chemistry, Materials and Chemical Engineering “Giulio Natta”, Politecnico di Milano, Milano, Italy

<sup>2</sup> Department of Mechanical Engineering, Politecnico di Milano, Milano, Italy

<sup>3</sup> Department of Materials and Mechanical Engineering, Centre for Advanced Steels Research, University of Oulu, Oulu, Finland

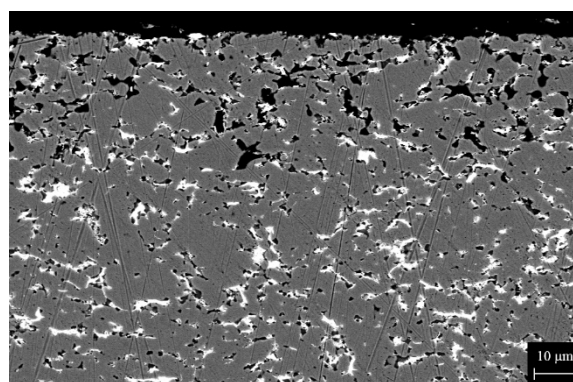
## Abstract:

The use of self-lubricating metal matrix composites undergoes an increasing interest in the last decades due to the wide range of applications: from the industry, to the automotive and aerospace fields. This class of materials allows to increase the efficiency of processes and machines, decreasing the wear of components, hence the time and costs due to maintenance, and the use of energy required to overcome friction and wear. This work analyses the influence of the operative conditions during the production of a copper-tungsten disulfide (Cu-WS<sub>2</sub>) composite via metal powder metallurgy, evaluating the effects on wear behavior. The powders follow a drying and ball milling steps to eliminate the moisture and humidity, then they are compressed by a defined pressure load and for a selected time span, and sintered. This study investigates the differences in the produced material due to the use of different pressure loads, i.e. 6 or 8 tonnes, different time spans for pressing, i.e. 5 or 15 minutes, and different sintering temperature, i.e. 550, 700, or 800 °C, evaluating different production paths.

The produced materials are characterized by density measurement, micro-indentation hardness and wear tests. The absence of unwanted third phases, that may originate from the sintering step and oxidation processes, is verified by X-ray diffractometry and Energy Dispersive X-Ray Analysis, and Scanning Electron Microscopy is used to identify the possible wear mechanisms involved. The effect of the different production parameters combinations is evaluated via wear test and the wear rate have been calculated by confocal laser scanning microscopy. The results highlight that the pressure load influence on wear behavior is higher than the one of the pressure time span, and increasing the load lower the wear rate. On the other hand, higher sintering temperatures determine the increase of porosity at the interface between the copper matrix and the solid lubricant, especially in the region close to the surface. This turns to worst wear behavior with the rise of delamination and debris formation. An optimum,

considering the selected operative condition, is reached for the highest pressure load, lower pressure time and lower sintering temperature.

**Keywords:** self-lubricating material, metal-matrix composite, wear, metal powder metallurgy, sintering



**Figure 1:** SEM picture of cross section of the sample Cu-WS<sub>2</sub> pressed by 8 tons for 5 minutes and sintered at 800 °C. Porosities (black) are clearly visible in the more superficial region and at the interface between the copper matrix (grey) and the tungsten disulfide (white) in the bulk.



# Fabrication of generated rough surface topographies through additive manufacture for replication and study in various polymer materials

J. Perris<sup>1</sup>, C. Kumar<sup>1</sup>, Y. Xu<sup>2</sup>, N. Gadegaard<sup>1</sup>, D. Mulvihill<sup>1</sup>

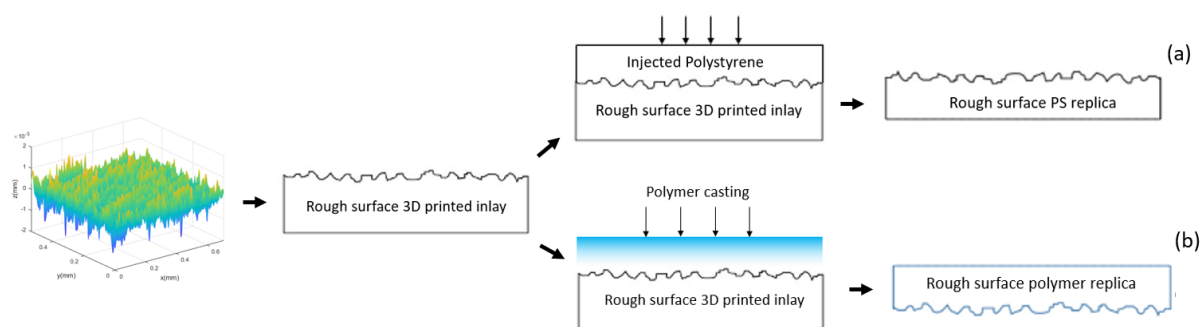
<sup>1</sup> James Watt School of Engineering, University of Glasgow, Glasgow, United Kingdom

<sup>2</sup> School of Mechanical Engineering, Hefei University of Technology, Hefei, China

## Abstract:

The work presented here introduces an Additive Manufacture (AM) based method that facilitates the production rough surface components from computationally designed topographies. The rough surface components were designed and then 3D printed using an SLA process. The AM components are utilised to produce rough surface topography replicas in various polymer materials. Two methods were employed to produce the topography replicas. Firstly, injection moulded replicas of the topographies were produced using the 3D printed components as the injection moulding inlay. This allowed the production of components in polystyrene (PS) and indicates the viability to produce high numbers of replica components in various thermoplastics. The second method involved the casting of low-viscosity polymer resins onto the 3D printed components. This method demonstrated the ability to produce high-resolution replicas of the topographies in various polymer materials. Two methods were used to quantify and validate the quality of surface replication, cross-covariance function ratio (ACCF) and a proprietary optical profilometry module designed for surface comparison. The results indicate high-quality surface replication in both processes. Injection moulding yields a high-throughput method with slightly reduced replication quality (ACCF = ~75%). While casting in low-viscosity polymer resins enables very high-quality replication (ACCF ranging from 75-90% in the different polymer resins) but a vastly reduced component production. Both methods yield highly promising results considering the complexity of the generated topographies [1]. The fabrication methods outlined here are aimed at allowing the generation and fabrication of varied topographies in a wide range of polymer materials for study and application within a research environment.

**Keywords:** Additive manufacture, microstructuring, replication, polymers, contact mechanics



**Figure 1** - Fabrication methods for generating polymer replicas of rough surface topographies (a) injection moulding fabrication route (b) polymer casting fabrication route.

## References:

1. Kumar C, Palacios A, Surapaneni VA, Bold G, Thielen M, Licht E, Higham TE, Speck T, Le Houérou V. Replicating the complexity of natural surfaces: technique validation and applications for biomimetics, ecology and evolution. *Philosophical Transactions of the Royal Society A*. 2019 Feb 11;377(2138):20180265.



# **SICT 2022 / Tribology 2022: i-Tribomat Workshop**

# i-TRIBOMAT – The Digital Service Provider for Tribological Materials up-scaling

Franz Pirker<sup>1</sup>

<sup>1</sup>. AC2T research GmbH, Wiener Neustadt, Austria

## 1. Introduction

The potential for saving energy and reducing costs [1] was the key reason to define tribology as its own scientific discipline. Fifty-five years after the birth of tribology, digitalisation and the Green Deal are the drivers and challenges at the forefront of industrial development. How can tribology position itself in the age of digitalisation? Which new digital tools and possibilities should be taken advantage of? What examples from other fields are there? MaaS (“Mobility as a Service”) is a good example of employing digital technology to develop a service that satisfies the needs of the population as well as the demands of resource efficiency and CO<sub>2</sub> reduction. Such services demand for an innovative digital business model and a flawless cooperation between the stakeholders. In the area of simulation and software, new business models and services are already well established – SaaS (“Software as a Service”) being a prime example. The trend goes away from one-time purchases towards needs-oriented usage and the suitable payment systems. With the European research project i-TRIBOMAT (“Intelligent Open Test Bed for Tribological Materials Characterisation”), the path towards TaaS – Tribology as a Service – is presented

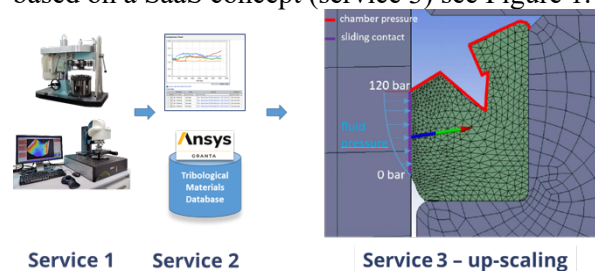
## 2. From Research Project to Service Provider

The EU-funded H2020 project i-TRIBOMAT develops new digital services which facilitate the rapid and cost-efficient selection of materials, as well as the prediction of the tribological performance of mechanical components. The project connects the entire tribological characterisation infrastructure of five leading European research centres and links it to an IT-platform using IoT technology. This allows the client to choose between over 100 different setups for material characterisation. The data is centrally stored and further processed in a newly developed cloud-based material database. The clients can access their data any time and can easily request an advanced analysis or create their own reports. Without needing a particular expertise, clients can carry out simulations in virtual workrooms, using their own material test results to rapidly and cost-efficiently predict their product’s performance without constructing a

prototype. All digital services can be customised and booked by the client on the web-based platform. The connection of infrastructures and the new digital services leads to Europe’s largest tribology centre. The European Tribology Centre (ETC) offers and markets all its services on a web-based platform.

## 3. Tribology as a Service – TaaS – The Platform

The European Tribology Centre’s core is a platform on which various services can be booked – from standardised tribometer tests and characterisation services (service 1) to data-driven services (service 2) and simulations in virtual workrooms based on a SaaS concept (service 3) see Figure 1.



**Figure 1:** Overview services and integrated workflow

All three services combined conform the integrated workflow to up-scale the material’s performance in a tribological component. If a customer wants to know how a new material would perform in his component under operation, the material would be tribologically tested, the test results and test data stored in a secure manner. Ultimately, the test results would be fed to tailored simulation models in a seamless workflow. These models up-scale the performance of the component using real test data as an input.

## 4. Conclusion

Digitisation is the opportunity and challenge that European industry must take on and utilise. You can’t think in terms of isolated methods and hype like digital twins or simulation, but you must build digital business models to offer added value to your customers. Integrated platforms such as i-TRIBOMAT show the how usually competing companies

can reposition themselves and thereby create a unique marketplace. Together we are stronger.

## 5. References

[1] K. Holmberg, A. Erdemir, Friction 5(3) 263 (2017)

## Acknowledgement

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No.

814494, project i-TRIBOMAT. More details:

<https://www.i-tribomat.eu/>.



European  
Commission

Horizon 2020  
European Union Funding  
for Research & Innovation



# On the challenges in achieving comparable and reproducible tribological model tests

M. Kröll <sup>1,\*</sup>, R. Grundtner <sup>2</sup>, K. Newrkla <sup>2</sup>, D. Spaltmann <sup>1</sup>, F. Pagano <sup>3</sup>, E. Nyberg <sup>4</sup>, V. Heino <sup>5</sup>

<sup>1</sup> Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany

<sup>2</sup> AC2T research GmbH, Wiener Neustadt, Austria

<sup>3</sup> Fundación Tekniker, Eibar Guipuzcoa, Spain

<sup>4</sup> Luleå Tekniska Universitet, Luleå, Sweden

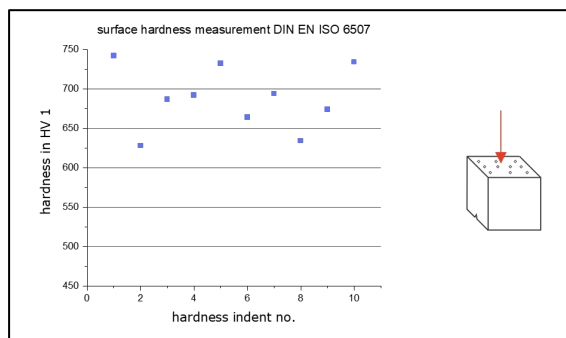
<sup>5</sup> Teknologian tutkimuskeskus VTT Oy, Espoo, Finland

## Abstract:

An infrastructure of more than 100 tribometers (for The European Tribology Centre i-TRIBOMAT) requires procedures which ensure that the respective results are characteristic as well as descriptive, trustworthy, comparable, and reproducible.

Many factors have an influence on the comparability and reproducibility of tribological data. It is therefore necessary to clarify the impact of these influences on the raw data as well as their processing and analyse the results, such as the coefficient of friction and wear volume.

The statement “garbage in, garbage out” does not only apply to numerical modelling. One of the most important input of tribological model tests are the samples used. Their composition, hardness and roughness predestine the performance of the tribological system. When performing round robin tests to assess the comparability of similar tribometers and set-ups, ideally identical samples are even more crucial. In addition, the respective properties have to be measured in a harmonised way.



**Figure 1:** An exemplary hardness distribution on the functional surface of a sample.

Samples, that are as identical as possible, and their characterisations are the first step towards comparable tribological model tests. However, further steps are required. Friction and wear are

often used to evaluate the performance of a tribological system. They suffer from the fact that different data processing methods exist that lead to incomparable results [1,2].

This lecture will show improvements achieved regarding the highlighted challenges.

## References:

1. I. Llavori, et al., Critical Analysis of Coefficient of Friction Derivation Methods for Fretting under Gross Slip Regime, Tribology International, Volume 143, 2020, 105988, ISSN 0301-679X, <https://doi.org/10.1016/j.triboint.2019.105988>.
2. J.J. Ayerdi, et al., Ball-on-flat linear reciprocating tests: Critical assessment of wear volume determination methods and suggested improvements for ASTM D7755 standard, Wear, Volumes 470–471, 2021, 203620, ISSN 0043-1648, <https://doi.org/10.1016/j.wear.2021.203620>.

## Acknowledgement:

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 814494, project i-TRIBOMAT. More details: <https://www.i-tribomat.eu/>.



European  
Commission

Horizon 2020  
European Union funding  
for Research & Innovation

# Digitalization of Tribological Systems for Decision-Making

Donna Dykeman<sup>1</sup>, Sara Onrubia<sup>1</sup>, Sunil Acharya<sup>2</sup>, Ulrike Cihak-Bayr<sup>3</sup>, Marin Herr<sup>3</sup>, Franz Pirker<sup>3</sup>, Dirk Spaltmann<sup>4</sup>, Mirco Kröll<sup>4</sup>

<sup>1</sup>Ansys UK Ltd., Materials Business Unit, Cambridge, United Kingdom

<sup>2</sup>Ansys Inc., Mechanical Business Unit, Canonsburg, United States

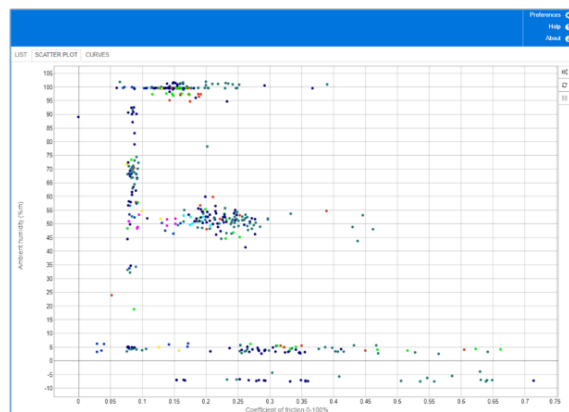
<sup>3</sup>AC2T Research GmbH, Wiener Neustadt, Austria

<sup>4</sup>Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany

## Abstract:

Introducing new materials and products to the market in any of the sectors of transportation, power generation or manufacturing, materials development and up-scaling requires a clear understanding of the materials tribological behaviour and durability under specific system operating conditions. This requires long-term tribological materials characterisation, large amounts of capital, and the availability of specific and often complex testing in different test scales. To reduce the risk, time and cost of research and industry investment, digitization of tribological systems and their characterization (physical and virtual) is of paramount importance for sharing knowledge, meeting safety and regulatory requirements, improving resource efficiency, and reducing the time-to-market. Due to the complexity of a tribological contact and diversity of characterization methods, the entire history of a sample, its respective counter-body and intermediate media, and environmental conditions needs to be captured for full traceability. The H2020 i-TRIBOMAT<sup>1</sup> project brings together experts in tribology characterization and modelling with leading materials information management technology, with one of the objectives being to establish best practices for digitalization of tribology characterization workflows for traceability and end-user decision-making. In this talk, we will explore the practicalities of establishing a materials information management system (Ansys Granta MI<sup>2</sup>) along with the business case<sup>3</sup> and best practices for digitalization for tribological systems to support the ongoing services of The European Tribology Center (ETC).

**Keywords:** tribology, digitalization, characterization, experimental, modelling, simulation, data, metadata, Ansys Granta MI, FAIR, Ansys Mechanical, up-scaling, business case



**Figure 1:** Tribology data for early-stage exploration and selection, illustrating the range of coefficients of friction (CoF) and the influence of humidity. This data provided by BAM is stored in the Ansys Granta MI materials information management system.

## References:

1. iTRIBOMAT Intelligent Open Test Bed for Materials Tribological Characterisation Services | [i-TRIBOMAT - Home](https://www.ansys.com/products/materials/granta-mi).
2. Ansys Granta MI | <https://www.ansys.com/products/materials/granta-mi>
3. Warde, S., Fairfull, A. 2020. The Business Case for Materials Intelligence. Ansys Inc. The Business Case for Material Intelligence | [Ansys White Paper](https://www.ansys.com/whitepapers/the-business-case-for-material-intelligence)

# From Characterisation Data to Material Product Performance a Seamless Workflow

U. Cihak-Bayr<sup>1\*</sup>

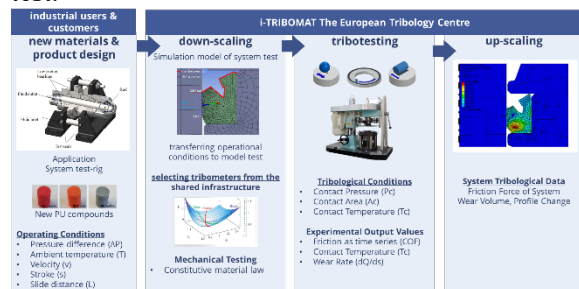
<sup>1</sup> AC2T research GmbH, Wiener Neustadt, Austria

## Abstract:

The design process of tribological systems involving new materials usually involve expensive, time-consuming experimental tests on full component scale. Within the European test-bed project i-TRIBOMAT („Intelligent Open Test Bed for Tribological Materials Characterisation“) we present an approach to upscale material performance and predict the product performance at an early development stage.

The upscaling tools in i-TRIBOMAT have been built based on the challenges of three use cases. Here, we illustrate the one on self-lubricating shaft seals operating in pneumatic cylinders.

Starting with customer requirements, the process of downscaling selects the appropriate model test and its parameter range. Downscaling is a complex process involving not only expertise knowledge on the specific application as well as on available tribological test-rigs, but also makes use of simulations. In the current use case, a FEM simulation of the mounting procedure provides the normal pressure ranges required in the model test.



**Figure 1:** Workflow for downscaling and up-scaling of material performance

A model test is a tribological test performed with small coupons of samples, easily produced for wide variation of materials, following a standardized procedures, to ensure full repeatability and robustness of the generated data. These data on friction and wear rate of the model-test experiment serve as input data for the upscaling simulation, which covers all the application specific geometries, loadings and kinematic conditions.

Due to the complexity of a tribological contact and diversity of characterization methods, not

only the test results have to be recorded in detail, but also the metadata of the samples and the tribological experiment, covering the entire history of a sample, its respective counter-body, intermediate media, and environmental conditions needs to ensure for full traceability. The database service of i-TRIBOMAT enables to search and select for the appropriate input data for the up-scaling simulation, following FAIR data standards.

The generated upscaling tools are universal in terms of flexible parametrization to other materials, here soft polymers such as other polyurethanes, and to comparable loading conditions leading to the same failure mechanism of the material.

As any simulation is just as good as its input data used, the centrepiece of the services provided by i-TRIBOMAT is the strong and seamless linkage of experimental data and simulation modelling activities. Trusted data are the basis for robust predictions on component level behaviour of materials regarding friction or wear or combination of both, thus enabling benchmark of materials with respect to life-time or efficiency of a component in a specific application.

**Keywords:** wear simulation, efficiency, seals, performance, trusted data, standardized tribological test.

## Acknowledgement

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 814494, project i-TRIBOMAT. More details: <https://www.i-tribomat.eu>





## Success story in developing seal materials for pneumatic systems

F. Pagano <sup>1\*</sup>, B. Pinedo <sup>1</sup>, A. Artetxe <sup>1</sup>, E. Rafols <sup>1</sup>, I. Minami <sup>2</sup>, L. Mont <sup>3</sup>

<sup>1</sup> Department of Tribology, Tekniker, Eibar, Spain

<sup>2</sup> Engineering Sciences and Mathematics, Lulea University of Technology, Lulea, Sweden

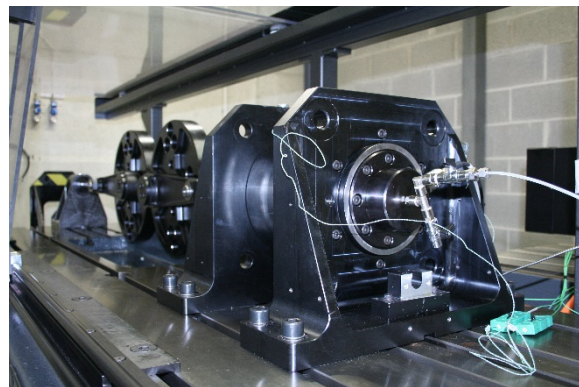
<sup>3</sup> Trygonal Iberia, Trygonal Group, Andoain, Spain

### Abstract:

Seals are interface substances that are placed between two different fluids (either liquids or gases) that should not contact each other. In practice, a seal isolates internal functional fluid from external environment, thereby preventing any contaminations from outside as well as spillage of the fluid to outside. The properties and performances of seal materials are key to maintain the system in ideal conditions. Since seal materials experience repeated mechanical stimuli such as load and shear stress, the robusticity against these stresses are essential. Although the material's tribological properties are of importance, practical materials are chosen empirically and the tribological analysis is not always fully considered. A sophisticated method which evaluates and helps to select proper materials for individual moving mechanical assembly (MMA) is highly desired by seal manufacturers and users. Under these circumstances, i-TRIBOMAT is challenging to develop a standard test method for seal materials, particularly those for hydraulic systems. For the i-TRIBOMAT purposes, TEKNIKER used TESSA (TEsting Seal-ing System Apparatus) to evaluate seal materials inhouse under the conditions closely related to the real operation parameters. It also validates the models, presented by AC2T in this session, thereby giving insight in the performance of the sealing systems. Concisely, TESSA is a versatile equipment which enables "upscaling protocol" for seal materials developed by i-TRIBOMAT consortium. Here "upscaling" stands for the prediction of the tribological performances in practice from the results with well-designed laboratory experiments within the strategy of "lab-to-field" approach. TESSA is an ideal equipment to bridge between laboratory and practice. It also provides the complementary relation between modeling and experiment. By interfacing to the i-TRIBOMAT digital tools, the utility of TESSA is further enhanced. In this way, i-TRIBOMAT well organizes three different phases in R&D – simple laboratory tests, in silico studies (modeling), and component test with TESSA – that are often conducted independently so far.

TESSA evaluates the effect of critical factors, such as pressure, velocity, mis-alignment, and temperature, on the performance of seal systems,

including lifetime prediction by accelerated tests. Two main modules, a reciprocating setup and a rotatory/oscillatory one, to test both reciprocating and rotatory seals. Both parts consist of an actuating system (electrical motor), a friction measurement device and a modular test chamber tailor made for each application. These specifications meet the requirements of the industrial partner, Trygonal who is manufacturing high quality plastic and sealing materials with a wide range of products and within advanced R&D strategy. Their presence in the international market has been dramatically growing in last decades. Polymeric lip seal materials newly developed by Trygonal were investigated in this work. The experimental part behind the success story will be presented herein.



**Figure 1:** Laboratory tribotest (up on the left), Simulation tool and TESSA test bench.

### References:

1. B. Pinedo, M. Conte, I. Perez, M. San Martin, E. Gomez-Acedo, A. Igartua. New high performance test rig for sealing systems characterization. World Tribology Congress 2013

### Acknowledgement:

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 814494, project i-TRIBOMAT. More details: <https://www.i-tribomat.eu/>.



European  
Commission

Horizon 2020  
European Union funding  
for Research & Innovation

# Game changing in material development through lab-to-field approach

Ichiro Minami

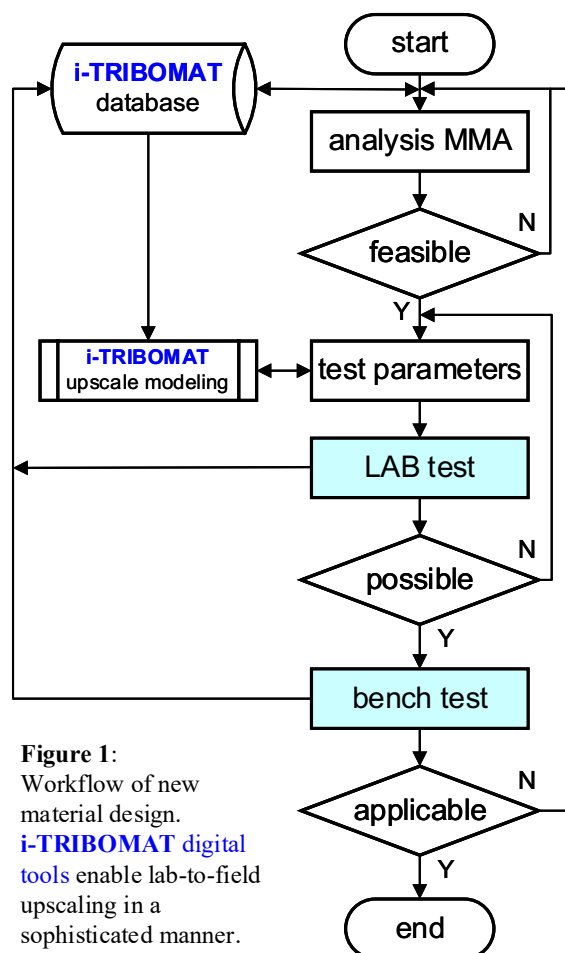
Luleå University of Technology, Luleå, Sweden

## Abstract:

Tribo-materials that construct moving mechanical assemblies (MMAs) must be robust as structural materials and should provide lubrication properties as functional materials. To date, ferrous materials are used in many MMAs with combination of proper lubricants. Updated technological and environmental requirements for MMAs arose the need of new tribology materials. To meet these demands, i-TRIBOMAT develops digital tools for material characterization and design particularly for tribology purposes [1]. This work presents how i-TRIBOMAT solves the difficulties in “lab-to-field” protocol in R&D of tribo-materials. Successful examples with use cases are also herein.

Historically, laboratory tribo-tests have been developed for trouble analysis of MMA operation, thereby trying to find a solution. Later, several standard tribo-tests have been authorized for quality control of lubricants. Nowadays lubrication engineers often employ these standard procedures in lubricant R&D. On the other hand, standardized protocol of designing tribo-material is scarcely found, even for screening purposes, so far. Since so many factors can/may influence the output signals, there remain uncertainty in applicability of the results, while it costs relatively high.

Figure 1 summarizes i-TRIBOMAT procedure. The work begins with setting the “lab-to-field” target in aid of i-TRIBOMAT digital tools. It follows to choose proper LAB tester and test matrix. After obtaining positive results from the LAB test, bench test (usually miniature sized practical MMA) will be conducted. Contrary to conventional trial & error procedure, i-TRIBOMAT prioritizes significant test parameters to maximize the time & cost efficiency in R&D. In addition, i-TRIBOMAT utilizes existing resources of tribo-testers owned by the consortium partners. More than 100 tribo-testers that covers all round tribology field are on standby. The digital tools well-manage these tribo-testers and tester and experiment setup will be offered properly for each task. As i-TRIBOMAT digital tools are continuously updating, the test results will be registered in the database for utilizing further material developments and characterizations.



**Figure 1:**  
Workflow of new material design.  
i-TRIBOMAT digital tools enable lab-to-field upscaling in a sophisticated manner.

## References:

1. F.Pirker, et al., i-TRIBOMAT: The Digital Service Provider for Tribological Materials upscaling, Tribologie+Schmierungstechnik, 67 (5-6), 35-50 (2020). DOI 10.30419/TuS-2020-0026

## Acknowledgement:

This project has received funding from the European Union’s Horizon 2020 research and innovation pro-gramme under grant agreement No. 814494, project i TRIBOMAT. More details: <https://www.i-tribomat.eu/>.



European  
Commission

Horizon 2020  
European Union funding  
for Research & Innovation

**SICT 2022 / Tribology 2022 joint  
Session II:  
Physics or chemistry of tribo-  
surfaces / Nanotribology (Part II)**

# Effect of humidity on capillary adhesion and friction at silicon-on-silicon multi-asperity interfaces

Liang Peng<sup>1</sup>, Feng-Chun Hsia<sup>1,2</sup>, Sander Woutersen<sup>1</sup>, Bart Weber<sup>1,2</sup>, Daniel Bonn<sup>1</sup>

<sup>1</sup> Van der Waals-Zeeman Institute, Institute of Physics, University of Amsterdam, Science Park 904, 1098 XH Amsterdam, The Netherlands

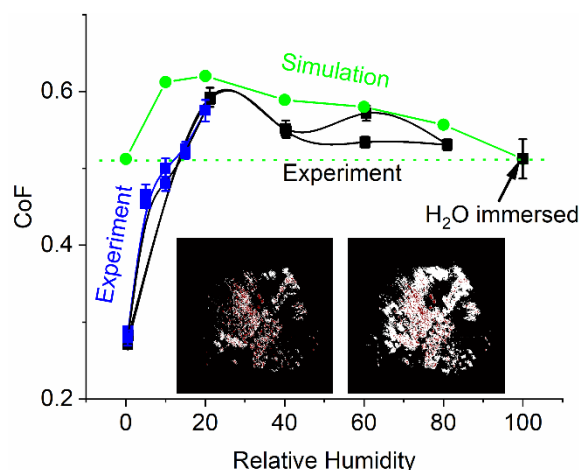
<sup>2</sup> Advanced Research Center for Nanolithography (ARCNL), Science Park 106, 1098 XG Amsterdam, The Netherlands

## Abstract:

Capillary adhesion can indirectly contribute to friction, especially at smooth interfaces in humid environments. We present how capillary adhesion and friction evolve with relative humidity at multi-asperity Si-on-Si friction interfaces. An adhesion model based on the boundary element method (BEM) is employed to reproduce the humidity-dependent friction behavior. It is found that the decrease of capillary adhesion with increasing relative humidity (20%-80%) leads to a reduction in friction. Replacing the water with isopropanol (IPA) at the interface can reduce the friction even further. The low friction in dry environments is attributed to the lack of both capillary adhesion and interfacial bonds. The contribution of interfacial hydrogen bonding to the friction is evidenced through heavy water immersed friction experiments.

white, and black regions in the contact map correspond to the contact area, capillary area, and non-contact area without water bridges, respectively.

**Keywords:** Capillary adhesion, friction, multi-asperity, bonding



**Figure 1:** Figure illustrating the evolution of Si-on-Si friction with relative humidity (RH). Measurements were performed with increasing and decreasing relative humidity to confirm that hysteresis was absent. The blue and black dashed lines indicate the experiment result, the simulation result is represented by the green dotted line. Inserts are two simulated contact maps under 20% RH (left) and 80% RH (right). The red,

# Frictional properties and nano-mechanical analysis of surface-attached hydrogels

R. Maraula<sup>1</sup>, J. R  he<sup>1</sup>

<sup>1</sup> Department of Microsystems Engineering (IMTEK), Albert Ludwig University, Freiburg, Germany

## Abstract:

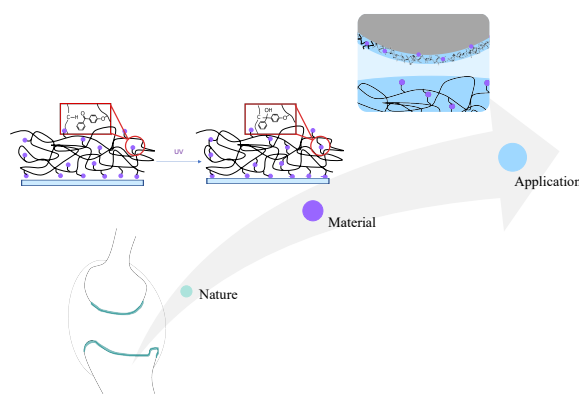
Tribological contacts account for about 23% of worldwide energy consumption. While 20% of this energy are used to overcome the friction, the 3% to repair wear failures. As a consequence, friction and wear cause costs of 250 billion euros every year and they are responsible for the emission of 8120 Mt of CO<sub>2</sub> [1].

This problem can be solved by creating materials which exhibit low friction, and therefore prevent the wear phenomena and minimize energy loss. Natural systems can be an inspiration for the development of technical systems because of their high efficiency, durability, and their ability to adapt themselves to new environmental situations. The evolutionary process has led to specialized surfaces where friction is adjusted to meet the requirement of the living organism for different purposes, for example the synovial joint of mammals, in which the synovial fluid plays a crucial role as water-based lubricant [2].

In this context, hydrogels are promising materials because they consist of a solid matrix, able to recover their initial shape after stress release, and a high content of water, similar to biological tissues.

The goal of this work is to investigate the mechanical behavior and frictional properties of hydrogels covalently attached to a rigid substrate (glass, silicon wafer) by C,H-Insertion Crosslinking (CHic) via UV radiation. CHic is a novel crosslinking method in which crosslinking and covalent attachment to the substrate occur simultaneously, as reactive groups are incorporated into polymer chains by a copolymerization reaction [3]. Moreover, it is important to investigate the role of the indenter geometry on these materials with nanoindentation experiments. When the surface-attached hydrogel is compressed, the polymer network undergoes conformational transitions between compressed and stretched chains and this is strongly dependent on the shape and size of the slider. As a consequence, a change in the geometry of slider (spheres of different radius, pyramid) might lead to different mechanical behavior.

**Keywords:** tribology, friction, wear, synovial joint, hydrogels, surface modification, CHic reaction, nanoindentation, contact mechanism, indenter geometry.



**Figure 1:** Figure illustrating the fundamental challenge of this project. Inspiration from the nature (synovial joint) leads to create new surface-attached hydrogels for CHic reaction. The final goal is to get low friction surface materials for any type of application.

## References:

1. Holmberg, K., Siilasto, R., Laitinen, T., Andersson, P., J  sberg, A. (2013), Global energy consumption due to friction in paper machines, *Tribology International*, 62, 58-77.
2. Mow, V. C., Ateschian, G. A., Spilker, R. L. (1993), Biomechanics of Diarthrodial Joints: A Review of Twenty Years of Progress, *J. Biomech. Eng.*, 115, 460-467.
3. Prucker, O., Naumann, C. A., R  he J. (1999), Photochemical Attachment of Polymer Films to Solid Surfaces via Monolayers of Benzophenone Derivatives Photochemical Attachment of Polymer Films to Solid Surfaces via Monolayers of Benzophenone Derivatives. *J. Am. Chem.*, 121, 38, 8766-8770.



# Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub> Nano-Sheets: Substrate-Dependent Tribo-Chemical Reactions

A. Rota<sup>1,2,3\*</sup>, N. Bellina<sup>3</sup>, A. Rosenkranz<sup>4</sup>

<sup>1</sup> Dipartimento di Scienze Fisiche, Informatiche e Matematiche – Università di Modena e Reggio Emilia, Modena, Italy

<sup>2</sup> CNR Nano – Istituto di Nanoscienze, Centro S3, Modena, Italy

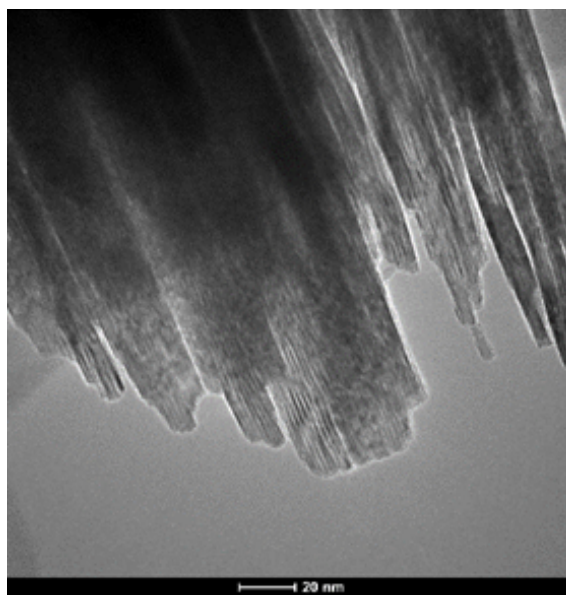
<sup>3</sup> Centro Interdipartimentale Intermech-Mo.Re., Università di Modena e Reggio Emilia, Modena, Italy

<sup>4</sup> Department of Chemical Engineering, Biotechnology and Materials, University of Chile, Santiago del Chile, Chile

## Abstract:

MXene nano-sheets are an emerging class of 2D materials composed by nitrides or carbides of transition metals, obtained by removing the A layers from their corresponding MAX phase. Among MXenes, Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub> represents a promising material for mechanical applications, related to its hardness, high melting point and good corrosion resistance, together with very good tribological properties due to the easy-to-shear ability between adjacent layers [1,2]. This work reports on the lubricating properties of Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub> ethanol suspension (1wt.-%) drop-casted on Fe and Cu substrates, tested in ball-on-disc configuration using an AISI-440C counter-body. For both substrates, the presence of the nano-sheets reduces friction, keeping the COF low and stable well after ethanol evaporation. However, in case of Cu, the effect is much longer compared to Fe, with a 35-fold increased lifetime. The analysis with Raman spectroscopy reveals very different scenarios for both substrates. In case of Fe, the progressive failure of MXene is due to the formation of oxides on both the substrate and the counter-body. Differently, in case of Cu, sliding induces the formation of a Ti<sub>3</sub>C<sub>2</sub>-based tribolayer on the substrate and steel ball, justifying the observed long-lasting effect. The observed tribological performance, together with their good mechanical properties, make Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub> nano-sheets an excellent candidate to be used as nano-additives for lubrication purposes. Further, this work aims at boosting theoretical and experimental studies on MXenes tribo-chemical processes.

**Keywords:** MXene nano-sheets, Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub>, Friction, Raman Spectroscopy.



**Figure 1:** Scanning electron micrographs of Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub> nano-sheets.

## References:

1. M. Naguib, M. et al., "Two-dimensional nanocrystals produced by exfoliation of Ti<sub>3</sub>AlC<sub>2</sub>" *Adv. Mater.* 23, 4248–4253, 2011.
2. Wyatt, B. C., Rosenkranz, A., & Anasori, B. (2021). 2D MXenes: Tunable Mechanical and Tribological Properties. *Advanced Materials*, 2007973.



# A Novel Restrictor Design for Hydrostatic Bearing

S. C. Lin<sup>1\*</sup>, Y. H. Lo<sup>1</sup>, Y. H. Lin<sup>1</sup>, W. T. Tung<sup>1</sup>, T. H. Lai<sup>2</sup>

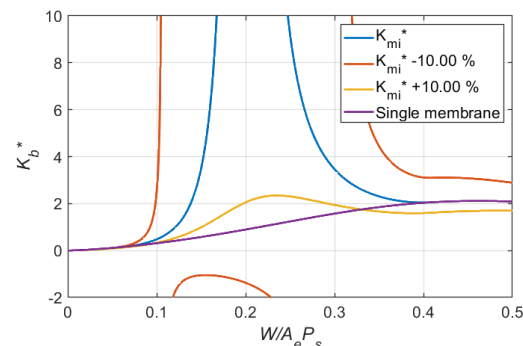
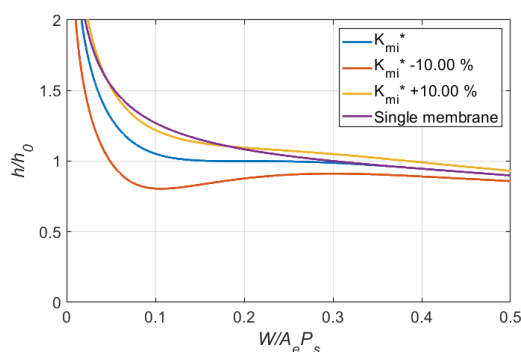
<sup>1</sup> Department of Power Mechanical Engineering, National Tsing Hua University, Hsin-Chu, Taiwan

<sup>2</sup> Department of Thermal Energy Conversion Technology, Industrial Technology Research Institute, Hsin-Chu, Taiwan

## Abstract:

It was known that a high static stiffness hydrostatic bearing system can be achieved as membrane restrictor are properly designed [1,2]. And the dimensionless stiffness of the membrane  $K_r^*$  and the design restriction ratio  $\lambda$  are the two key parameters for membrane restrictor design. It was found theoretically that high bearing stiffness can be achieved within some loading region (i.e.  $W/A_e P_s = 0.2 - 0.5$ ). However, the stiffness of the bearing is very low in light loading region. In this study, a new design of restrictor was proposed. A series of simulations were conducted to simulate system performance. The main advantages of the proposed restrictor are increasing the flexibility of providing high stiffness at desired loading region; and improving the stiffness performance of the bearing system at light loading region. In order to verify the performance of the proposed design, a series of experiments were conducted. It was shown that the test results were quite close to the simulation results for bearing compensating with both single membrane and dual membrane restrictors. And as expected, the proposed dual membrane restrictor can improve the performance of the bearing in light loading region.

**Keywords:** Diaphragm controlled restrictor, Membrane restrictor, Hydrostatic bearing



**Figure 1:** Figure illustrating the comparison between single membrane restrictor and the proposed design on clearance ratio and static stiffness of the compensated hydrostatic bearing.

## References:

1. Lai, T.H., Chang, T.Y., Yang, Y.L., and Lin S.C., (2017) Parameters design of a membrane-type restrictor with single-pad hydrostatic bearing to achieve high static stiffness, *Tribology International*, 107, 206-212.
2. Lai, T.H. and Lin, S.C., (2018) A simulation study for the design of membrane restrictor in an opposed-pad hydrostatic bearing to achieve high static stiffness, *Lubricants*, 6(3), 71.

# Skin friction: mechanical and tribological characterization of different papers used in everyday life

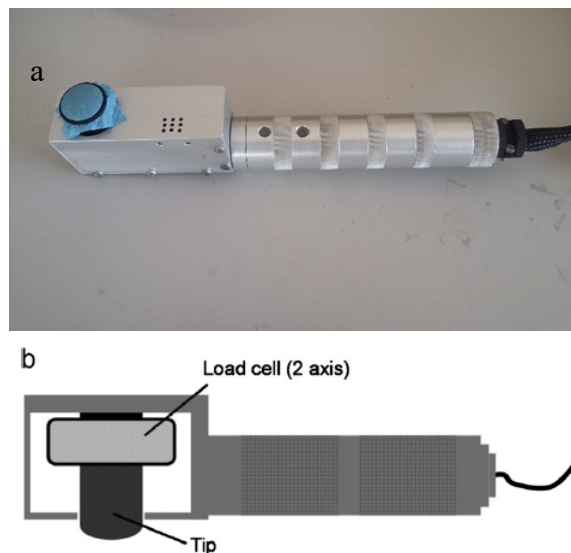
L.M. Vilhena<sup>1\*</sup>, A. Ramalho<sup>1</sup>

<sup>1</sup> CEMMPRE Centre for Mechanical Engineering Materials and Processes, University of Coimbra, Coimbra, Portugal

## Abstract:

The degree of comfort or the degree of discomfort is often incorporated in the materials selection by manufacturers of different skin-product interactions. According with recent studies, soft and smooth materials are pleasant, and those that were stiff, rough, or coarse are unpleasant [1]. Analysis of comfort and discomfort in such a products that involve sliding, reveals the relative importance of skin friction as well the deformation of the skin during sliding. In fact, the exploratory procedure that is used to touch a surface is similar to determining friction in a tribological test, since by pressing your skin at the sliding surface and feel specific features (touch perception), friction is generated in the contact. The coefficient of friction for different contacting materials against skin is mainly influenced by the nature of the materials (synthetic and natural fabrics), mechanical contact parameters (interfacial pressure and sliding velocities), and physiological skin conditions (ambient humidity and skin moisture content) [2]. In the present research work, 7 different types of paper used in everyday life were analysed. The physical properties of this materials were determined, through tensile tests and friction tests. After that, tactile perception questionnaires were performed, where 29 volunteers assessed the different types of paper on a 1 to 5 haptic scale. By comparing physical properties with tactile qualities, it was possible to conclude that the coefficient of friction is strongly correlated with the sensations of softness and pleasant touch.

**Keywords:** human skin, coefficient of friction, paper, mechanical properties, relative humidity



**Figure 1:** Portable measuring probe to measure skin friction: (a) picture showing the O-ring and a type of paper and, (b) schematic picture of the multi-component force sensor.

## References:

1. Ramalho, A., Szekeres, P. e Fernandes, E. (2013), «Friction and tactile perception of textile fabrics», *Tribol. Int.*, volume 63, 29–33.
2. Vilhena, L. e Ramalho, A. (2016), «Friction of Human Skin against Different Fabrics for Medical Use», *Lubricants*, 4 (1) 6, 1-10.

## **SICT Session II: Coatings for Energy and Environmental Applications**

# Optical design, microstructural characterization and high-temperature in-air stability study of solar selective coatings based on aluminium-(titanium, chromium) oxynitride multilayers

R. Escobar Galindo<sup>1\*</sup>, I. Heras<sup>2</sup>, E. Guillén<sup>3</sup>, F. Munnik<sup>4</sup>, I. Azkona<sup>5</sup>, A. Caro<sup>6</sup>, T.C. Rojas<sup>6</sup>, J.C. Sánchez-López<sup>6</sup>, M. Krause<sup>4</sup>

<sup>1</sup> Departamento de Física Aplicada I, Escuela Politécnica Superior, Universidad de Sevilla, Sevilla, Spain

<sup>2</sup> Advanced Center for Aerospace Technologies (CATEC), Sevilla, Spain

<sup>3</sup> Profactor GmbH, Steyr-Gleink, Austria

<sup>4</sup> Helmholtz-Zentrum Dresden-Rossendorf, Dresden, Germany

<sup>5</sup> Metal Estalki S.L., Zamudio, Spain

<sup>6</sup> Instituto de Ciencia de Materiales de Sevilla (CSIC-Univ. Sevilla), Sevilla, Spain

## Abstract:

The development of new solar selective coatings (SSCs) operating in air at high temperatures is an actual challenge for the development of Generation 3 concentrated solar power (CSP) plants. In particular, current central tower systems operate at maximum temperatures of 550 °C mainly due to the severe degradation that the state of the art absorber paints (i.e. Pyromark®) suffer at higher temperatures. Aluminium metal oxynitrides  $\text{Al}_y\text{Me}_{1-y}\text{O}_x\text{N}_{1-x}$  (Me = Ti, Cr) prepared by physical vapour deposition (i.e. cathodic vacuum arc and HiPIMS) were selected as candidate materials for SSCs on the basis of stability considerations of the tentatively formed nitrides and oxides. The optical properties of these films can be controlled in a wide range from a metallic to a dielectric character by varying the oxygen and nitrogen content.

In the last years we have performed an extensive research on the design, fabrication and high-T exposure of SSCs based on aluminium-titanium [1,2] and aluminium-chromium oxynitrides [3]. Once single thin films were fully characterized by ion beam analysis, scanning and transmission electron microscopy and X-ray diffraction, complete SSCs were designed with optical simulations, based on measured optical constants of each of the individual layers, providing excellent optical selective properties in terms of solar absorptance ( $\alpha$ ) and thermal emittance ( $\epsilon_{RT}$ ). The selected multilayers stacks were deposited, obtaining excellent agreement between simulated and experimental reflectance spectra. Finally, the thermal stability in air of the complete deposited SSCs was analyzed by isothermal and cyclic heating tests simulating operating conditions.  $\text{Al}_y\text{Ti}_{1-y}(\text{O}_x\text{N}_{1-x})$  SSCs showed no degradation after 750h of cycles in air at 600°C and these results were compared with *in-situ* high temperature an-

nealing performed in vacuum at the multi-chamber cluster tool situated at the Helmholtz-Zentrum Dresden-Rossendorf (HZDR) [4], confirming that these stacks withstand breakdown at 600°C in air and 800°C in vacuum.  $\text{Al}_y\text{Cr}_{1-y}(\text{O}_x\text{N}_{1-x})$  SSCs stacks presented a good solar selectivity with  $\alpha > 95\%$  and  $\epsilon_{RT} < 15\%$ , and fulfilled the performance criterion after 600 and 700 °C short term heating treatments. At 800 °C, they underwent a further structural transformation, provoked by the oxidation of the inner layers, and they consequently lost their solar selectivity.

In this invited talk, we will summarize these results and present current research strategies to further improve the performance of the developed materials.

**Keywords:** solar selective coatings, thermosolar energy, PVD, thin films, in-situ characterization.

## References:

1. I.Heras, E. Guillén, F. Lungwitz, G. Rincón-Llorente, F. Munnik, E. Schumann, I. Azkona, M. Krause, R. Escobar-Galindo. Sol. Energy Mater. Sol. Cells 176 (2018) 81-92.
2. R. Escobar-Galindo, E. Guillén, I. Heras, G. Rincón-Llorente, M. Alcón-Camas, F. Lungwitz, F. Munnik, E. Schumann, I. Azkona, M. Krause. Sol. Energy Mater. Sol. Cells 185 (2018) 183-191.
3. T.C. Rojas Ruiz, A. Caro, R. Escobar-Galindo, J.C. Sanchez Lopez. High-temperature solar-selective coatings based on Cr(Al)N. Part 2: Design, spectral properties and thermal stability of multilayer stacks. Sol. Energy Mater. Sol. Cells. 218 (2020) 110812
4. R. Wenisch, F. Lungwitz, D. Hanf, R. Heller, J. Zscharschuch, R. Hübner, J. von Borany, G. Abrasonis, S. Gemming, R. Escobar-Galindo, M. Krause. Anal. Chem. 90 (13) (2018) 7837-7842.

# SYNTHESIS OF GRAPHENE-SUSPENSIONS BY PLASMA-IN-LIQUID PROCESS FOR THIN FILM COATINGS OF POLYMER-MEMBRANES FOR FUEL CELL APPLICATION

C. Rojas <sup>1</sup>, T. Schulz <sup>1</sup>, F. Käufer <sup>1</sup>, P. Quarz <sup>3</sup>, V. Bravo <sup>3</sup>, J. Kapp <sup>4</sup>, V. Lukassek <sup>4</sup>, T. Sommer <sup>5</sup>, P. Scharfer <sup>3</sup>, C. Scheu <sup>2</sup>, J. Wartmann <sup>4</sup>, W. Schabel <sup>3</sup>, A. Kruth <sup>1</sup>

<sup>1</sup> Leibniz Institute For Plasma Science and Technologie, Greifswald, Germany

<sup>2</sup> Max-Planck-Institut für Eisenforschung, Düsseldorf, Germany

<sup>3</sup> Thin Film Technology (TFT), Karlsruhe Institute of Technology (KIT), Karlsruhe, Germany

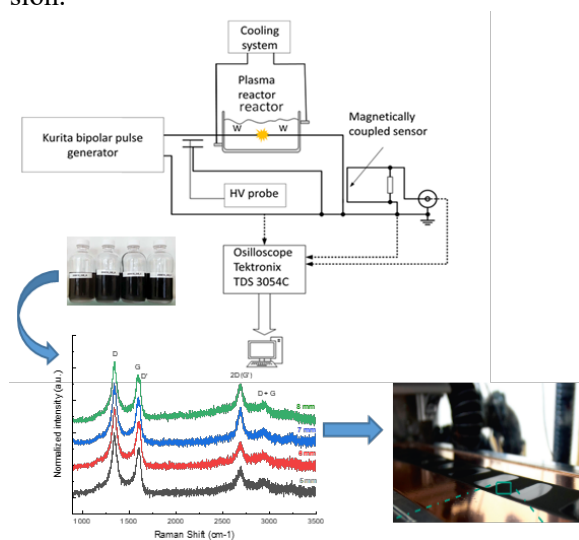
<sup>4</sup> Hydrogen and fuel cell center ZBT GmbH

<sup>5</sup> Plastic center-SKZ, Wuerzburg, Germany

## Abstract:

Over the past years, the need for graphene-based nanomaterials for new energy storage and conversion technologies is rapidly growing. In the new concepts for membrane electrode assemblies for polymer exchange fuel cells (PEMFCs), graphene may be used for coating of proton conductive membranes to prevent permeation of undesired species such as methanol and improve selectivity of the electrolyte. However, availability of cost-efficient synthesis processes to obtain high-quality graphene for coating of Nafion in large-scale membrane production is still a challenge. Recently, plasma in liquid (PiL) synthesis processes are developed as eco-friendly rapid single-step procedures to obtain ready-to-use nanomaterial suspensions, possessing a tremendous potential to increase performance and cost-efficiency of manufacturing for nanomaterials [1]. In PiL processes, synthesis reactions occur over comparably very short processing times such as minutes whilst achieving favorable properties such as high crystallinity and purity of nanoparticle suspensions. [2]. This paper presents a new PiL route to produce graphene suspensions. Graphene production is achieved via plasma-induced hydrocarbon cracking using ethanol as a precursor [3]. The experimental set-up is based on a rod-to-rod cell configuration using tungsten electrodes and applying pulsed DC voltage, 300 kHz frequency, and short pulse length within the ns range. Structural characterization of the obtained product by electron microscopy, X-Ray Photoelectron Spectroscopy and Raman spectroscopy revealed two-layer-graphene. Byproducts such as phenylethylene, styrene, and naphthalene were obtained to be in the ppm range by mass spectroscopy. Characterisation of formulated suspensions showed high stability over the duration of 90 days. Suspensions were found suitable for coating of Nafion membranes and application as GraphenBlocker-electrolytes in membrane electrode assemblies for direct methanol fuel cells.

**Keywords:** Plasma-in-liquid processing, coating technologies, proton conducting membranes, polymer exchange fuel cells, graphene suspension.



**Figure 1:** Schematic image of synthesis of PiL method using ethanol for coating production

## References:

1. Mun, M. K.; Lee, W. O.; Park, J. W.; Kim, D. S.; Yeom, G. Y.; Kim, D. W., Nanoparticles synthesis and modification using solution plasma process. *Applied Science and Convergence*.
2. Saito, G.; Akiyama, T., Nanomaterial Synthesis Using Plasma Generation in Liquid. *Journal of Nanomaterials* 2015, 2015, 123696.
3. Hagin Saito, G.; Akiyama, T., Nanomaterial Synthesis Using Plasma Generation in Liquid. *Journal of Nanomaterials* 2015, 2015, 123696.o, T., et al. (2012).

# Impact of Crosslinker Chemistry and Concentration on Reinforcement of GO Composites for Water Filtration Applications

P. Kaur<sup>1</sup>, R.L. Thompson<sup>1\*</sup>, L.R. Hutchings<sup>1</sup>, M.U. Chaudhary<sup>2</sup> and T. Pugh<sup>3</sup>

<sup>1</sup>Department of Chemistry, Durham University, Durham, United Kingdom

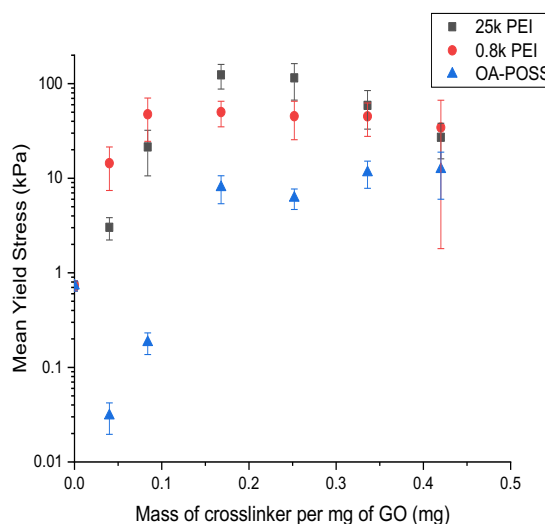
<sup>2</sup>Department of Engineering, Durham University, Durham, United Kingdom

<sup>3</sup>Evove, Sci Tech Park, Daresbury, United Kingdom

## Abstract:

Crosslinkers are important for GO plates in filtration applications because they help to define and maintain the integrity of the nanoscale channels. The rheological behaviour of GO based gels was studied to evaluate the effectiveness of three different types of crosslinkers. The  $G'/G''$  crossover yield stress values were used to estimate the ability of GO based gel membranes to withstand pressures applied in water filtration applications. Analysis of amplitude sweep rheology data for various concentrations of OA-POSS (rigid cage), as well as short ( $M_w = 0.8k$ ) and long ( $M_w = 25k$ ) chain branched PEI (flexible) crosslinkers, shows that crosslinking with either of two PEI polymers increases the yield stress of GO membranes up to 20 times more than the OA-POSS crosslinker. Overall, the addition of all three crosslinkers tested, significantly increases the elastic modulus of the GO membranes, compared to uncrosslinked GO. This increase in the elastic modulus is significant in ensuring the membranes resist distortion under pressure. Membranes crosslinked *in-situ* are swollen with water. A further investigation was carried out to study the effect of compression of a PEI crosslinked membrane by decreasing the distance between the geometry head and sample plate. This shows an increase in the elastic modulus of the PEI crosslinked membrane by 150% within the ranges tested due to the selective exclusion of water from the composite under compression. The yield stress of all three types of composite membranes increases up to a certain concentration of the crosslinker, before decreasing again which may be attributed to the saturation of available sites on GO nanosheets inhibiting crosslinking.

**Keywords:** graphene oxide (GO), yield stress, filtration, octaammonium polyhedral oligomeric silsesquioxane (OA-POSS), polyethyleneimine (PEI), elastic modulus, nanosheets



**Figure 1:** Graph showing the change in  $G'/G''$  crossover yield stress values for crosslinked GO membranes with increasing concentrations of the three different types of crosslinker.

## References:

1. Parsamehr, P. S., Zahed, M., Tofighy, M. A., Mohammadi, T. & Rezakazemi, M. (2019). Preparation of novel cross-linked graphene oxide membrane for desalination applications using (EDC and NHS)-activated graphene oxide and PEI. *Desalination* 468, 114079.
2. Yang, L., Liu, X., Wu, H., Wang, S., Liang, X., Ma, L., Ren, Y., Wu, Y., Liu, Y., Sun, M., & Jiang, Z. (2019). Amino-functionalized POSS nanocage intercalated graphene oxide membranes for efficient biogas upgrading. *Journal of Membrane Science* 596, 117733.



## **Plasma Tech Session IV: Plasma application in Energy and Environment**

# Validation of complex plasma chemistries: CO<sub>2</sub> as a case study

V. Guerra<sup>1</sup>, T. Silva<sup>1</sup>, C. Fromentin<sup>1</sup>, T. C. Dias<sup>1</sup>, A. S. Morillo-Candas<sup>2,3</sup> and O. Guaitella<sup>2</sup>

<sup>1</sup>Instituto de Plasmas e Fusão Nuclear, Instituto Superior Técnico, Universidade de Lisboa, Portugal

<sup>2</sup>Laboratoire de Physique des Plasmas (UMR 7648), CNRS, Univ. Paris Saclay, Sorbonne Université, École Polytechnique, France

<sup>3</sup>Paul Scherrer Institute, 5232 Villigen PSI, Switzerland

## Abstract:

Low-temperature molecular plasmas are complex systems, where energetic electrons initiate a rich non-equilibrium chemistry by transferring their energy to the heavy-particles in a variety of collisional processes, that include excitation of vibration and electronic states, dissociation, and ionization. The presence of different particles, with specific properties and energies, makes these plasmas useful in a wealth of applications, ranging from aerospace to nanotechnologies. However, the different kinetics become strongly coupled and difficult to disentangle, so that it is often difficult to identify the dominant phenomena and to optimize the plasma source for a particular application. Modelling emerges as a powerful tool, both to qualitatively interpret physical mechanisms and to quantitatively make predictions for real applications, ultimately leading to a better understanding of the relevant processes and input data. A key element is model *validation*, i.e., the comparison of simulations with experimental results or observations.

In recent years, several laboratories in Portugal, France, The Netherlands, and Russia have engaged in a collective effort to systematically pursue a strategy for the development of kinetic models and validation of plasma chemistry schemes in molecular plasmas. CO<sub>2</sub> plasmas were chosen as a case-study, due to their importance in applications and the inherent coupling of electron, vibration, chemical, ion and surface kinetics [1]. The approach focuses on the design of experiments where specific aspects of the plasma kinetics can be isolated, providing the ideal testbed for validation of a particular aspects of the kinetics and the associated collisional data. Such step-by-step validation procedure has already allowed the validation of: complete and consistent cross section sets for CO<sub>2</sub> [2] and CO [3,4]; vibration-translation (V-T) and vibration-vibration (V-V) energy exchanges [5], and electron-vibration (e-V) input of vibrational energy [6] in low-excitation conditions; atomic oxygen recombination kinetics [7,8]; the electron impact dissociation cross section [9]; dynamics of gas heating in the afterglow [10]; back reaction

mechanisms at low pressure [11,12]; chemical kinetics in vibrationally-cold plasmas [13]; and plasma chemistry in vibrationally excited plasmas [14,15]. The extension to other gases and mixtures will also be discussed at the conference.

**Acknowledgements:** This work was partially supported by the European Union's Horizon 2020 research and innovation programme under grant agreement MSCA ITN 813393, and by Portuguese FCT-Fundação para a Ciência e a Tecnologia, under projects UIDB/50010/2020, UIDP/50010/2020 and PTDC/FIS-PLA/1616/2021.

**Keywords:** CO<sub>2</sub> recycling, molecular plasmas, modelling, validation, reaction mechanism.

## References:

1. L. D. Pietanza *et al* (2021) *Eur. Phys. J. D* 75, 237.
2. M. Grofulović *et al* (2016) *J. Phys. D: Appl. Phys.* 49, 395207.
3. P. Ogloblina *et al* (2020) *Plasma Sources Sci. Technol.* 29, 015002.
4. L. Vialetto *et al* (2021) *Plasma Sources Sci. Technol.* 30, 075001.
5. T. Silva *et al* (2018) *Plasma Sources Sci. Technol.* 27, 015019.
6. M. Grofulović *et al* (2018) *Plasma Sources Sci. Technol.* 27, 115009.
7. J. P. Booth *et al* (2019) *Plasma Sources Sci. Technol.* 28, 055005.
8. A. S. Morillo-Candas *et al* 2019 *Plasma Sources Sci. Technol.* 28, 075010.
9. A. S. Morillo-Candas *et al* (2020) *Plasma Sources Sci. Technol.* 29, 01LT01.
10. T. Silva *et al* (2020) *Plasma Chem. Plasma Process.* 40, 713.
11. A. S. Morillo-Candas *et al* (2020) *J. Phys. Chem. C* 124, 17459.
12. T. Silva *et al* (2021) *J. CO<sub>2</sub> Utiliz.* 53, 101719.
13. A. F. Silva *et al* (2020) *Plasma Sources Sci. Technol.* 29, 125020.
14. P. Ogloblina *et al* (2021) *Plasma Sources Sci. Technol.* 30, 065005.
15. C. Fromentin *et al*, in preparation.

# Plasma-ammonia formation integrating high-temperature hydrogen plasma formation and biomass gasification

Nguyen Van Duc Long<sup>1,2</sup>, Kevin van 't Veer<sup>3</sup>, Jose Osorio Tejada<sup>1</sup>, Nam N. Tran<sup>2,4</sup>,  
Laurent Fulcheri<sup>5</sup>, Bhaskar Patil<sup>6</sup>, Volker Hessel<sup>1,2\*</sup>, Annemie Bogaerts<sup>3\*</sup>

<sup>1</sup>School of Engineering, University of Warwick, United Kingdom

<sup>2</sup>School of Chemical Engineering and Advanced Materials, University of Adelaide, Australia

<sup>3</sup>Department of Chemistry, Research Group PLASMANT, University of Antwerp, Universiteitsplein 1, 2610 Antwerp, Belgium

<sup>4</sup>Department of Chemical Engineering, Can Tho University, Vietnam

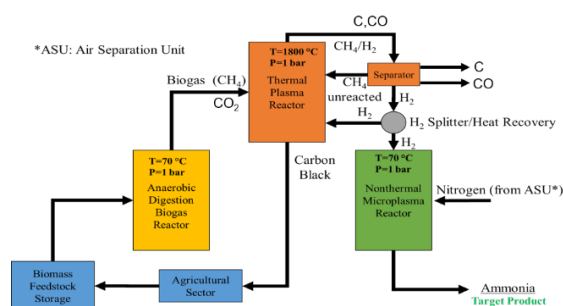
<sup>5</sup>MINES ParisTech, PSL-Research University, PERSEE, Sophia-Antipolis, France

<sup>6</sup>Nouryon, Capability Group, Process Technology - Reaction Engineering, Deventer/the Netherlands

## Abstract:

To reduce the fossil-fuel consumption and the impacts of conventional ammonia production on climate change, green ammonia production processes using green hydrogen and reaction intensification need to be investigated. Therefore, this work proposes a novel thermally integrated process design for the connected  $H_2$  production from biogas and  $NH_3$  synthesis by means of plasma technology. Raising the temperature of the non-thermal plasma (NTP) process, which is typically below  $100^\circ C$ , can be achieved by taking advantage of the heat released from the high temperature plasma (HTP) process. The integrated process was modelled and chemical kinetics simulations in the NTP section was conducted to determine the thermodynamically feasible operating window of this novel combined plasma process. Results suggest that an  $NH_3$  yield of 2.2% can be achieved at  $302^\circ C$  at an energy yield of  $1.1 \text{ g } NH_3/kWh$ . Cost calculations show that the economic performance is far from commercial, mainly because of the too low energy yield of the NTP process. However, when our costs are based on the best literature value and plausible future scenarios for the NTP energy yield, a cost prediction of below  $463\$/\text{ton } NH_3$  can be reached, which is competitive with conventional small-scale Haber-Bosch  $NH_3$  synthesis for distributed production. In addition, it is demonstrated that biogas can be used as feed, thus allowing the proposed integrated reactor concept being part of a biogas-to-ammonia circular concept.

**Keywords:** ammonia synthesis, biogas, nonthermal plasma, high temperature plasma, integration.



**Figure 1:** Schematic illustration of the pathway for the conversion of biomass to fertiliser using anaerobic digestion, thermal plasma, and non-thermal plasma.

## References:

1. Sarafranz, M. M.; Tran, N. N.; Nguyen, H.; Fulcheri, L.; Burton, R.; Wadewitz, P.; Butler, G.; Kirton, L.; Hessel, V. Tri-fold Process Integration Leveraging High- and Low-temperature Plasmas: From Biomass to Fertilizers with Local Energy and for Local Use. *J. Adv. Manuf. Process.* 2021, 3 (2), 1–21.
2. van 't Veer, K.; Reniers, F.; Bogaerts, A. Zero-Dimensional Modelling of Unpacked and Packed Bed Dielectric Barrier Discharges: The Role of Vibrational Kinetics in Ammonia Synthesis. *Plasma Sources Sci. Technol.* 2020, 29 (4), 045020.

## Plasma-catalytic coupling in a nanosecond pulsed discharge plasma for CO<sub>2</sub> recycling

M. Faedda <sup>1,2,\*</sup>, B. Samojeden <sup>1</sup>, L.M. Martini <sup>2</sup>, M. Motak <sup>1</sup>, P. Tosi <sup>2</sup>

<sup>1</sup> Department of Fuel Technologies, AGH University of Science and Technology, Cracow, Poland

<sup>2</sup> Department of Physics, University of Trento, Trento, Italy

### Abstract:

Carbon Capture, Storage and Utilization (CCSU) technologies can help us tackle the problem of cutting down greenhouse gas emissions. With CCSU the chemical processing of CO<sub>2</sub> treats this compound not as a harmful pollutant, but as a source of carbon that can eventually lead to value added products. Dry Reforming of Methane (DRM) is, in this context, already an established process for the conversion of carbon dioxide and methane into synthesis gas, even though it still struggles to become a mature industrial technology. Thermal catalytic DRM is the most widespread process <sup>1</sup>. However, plasma-activated and plasma-catalytic DRM underwent an increased interest in the recent years, due to improved efficiency and performances. Nevertheless, the interaction between unconventional types of plasmas and catalysts is still an unexplored field. In our experiments, the chosen plasma discharge is a nanosecond pulsed discharge plasma (NRP) operated at atmospheric pressure, which has been characterized in the DRM reaction by Scapinello and Montesano<sup>2,3</sup>. NRP is gaining growing attention as one of the most energy-efficient methods to promote chemical reactions, by taking advantage of the high electron densities and electron energies that can be produced. At the same time, due to the high temperatures reached, the coupling with a catalyst is only possible in a post-plasma catalysis configuration, where the risk of non-activation of the catalyst by the plasma is proportional to their distance. In our work, we synthesize a ceramic monolith foam catalyst that surrounds the discharge in a co-axial geometry from a close distance. The enhancement in the reactants conversion and influence on the products selectivities is studied as a function of different parameters, such as pore density, support material and active phase choice.

**Keywords:** plasma-catalysis synergy, post-plasma catalysis, nanosecond pulsed discharge, methane dry reforming, Ni-based catalyst, monolithic catalyst, porous catalyst, foam replica method, ceramic catalyst.

### References:

1. Świrk, K. *et al.* Yttrium promoted Ni-based double-layered hydroxides for dry methane reforming. *J. CO<sub>2</sub> Util.* **27**, 247–258 (2018).
2. Scapinello, M., Martini, L. M., Dilecce, G. & Tosi, P. Conversion of CH<sub>4</sub>/CO<sub>2</sub> by a nanosecond repetitively pulsed discharge. *J. Phys. D. Appl. Phys.* **49**, (2016).
3. Montesano, C., Faedda, M., Martini, L. M., Dilecce, G. & Tosi, P. CH<sub>4</sub> reforming with CO<sub>2</sub> in a nanosecond pulsed discharge. The importance of the pulse sequence. *J. CO<sub>2</sub> Util.* **49**, 101556 (2021).

# Effect of morphology of nanostructured CeO<sub>2</sub> supports on plasma-assisted CO<sub>2</sub> methanation

B. Musig<sup>1,2</sup>, M.E. Gálvez<sup>2</sup>, M.V. Navarro<sup>1</sup>

<sup>1</sup> Instituto de Carboquímica (CSIC), Zaragoza, Spain

<sup>2</sup> Institut Jean le Rond d'Alembert, CNRS UMR 7190, Sorbonne Université, Paris, France

## Abstract:

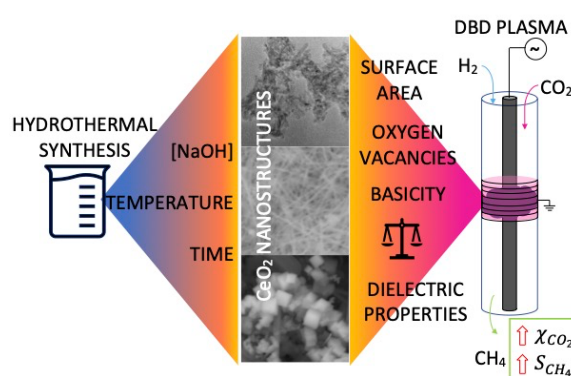
CO<sub>2</sub> methanation, i.e. through the Sabatier's reaction, is a promising technology for the valorization of CO<sub>2</sub>. It allows both the synthesis of a carbon-neutral fuel and the storage of off-peak renewable electricity (power-to-gas concept). The Sabatier's reaction is exothermic but strongly hindered by kinetics. It has been recently demonstrated that the use of a non-thermal plasma in combination with a Ni-containing catalysts results in an enhanced reaction yield even at atmospheric pressure and mild reaction temperatures (around 200°C)<sup>1</sup>. Plasma-catalyst coupling still represents one of the biggest challenges. Tailoring and optimizing the physico-chemical and electro-chemical properties of catalytic materials can lead to improved catalytic performance and lower energy consumption.

In the present study, different nanostructured CeO<sub>2</sub> supports were prepared for the Ni catalysts, to be used for CO<sub>2</sub> methanation in a dielectric barrier discharge (DBD) reactor, with focus on the contribution of CeO<sub>2</sub> morphology on the properties of the catalysts and their performance. The morphologies such as polyhedral nanoparticles, nanorods or nanocubes were tuned through the modification of their hydrothermal synthesis conditions, following a design of experiments (DOE) procedure. Subsequently, the supports were wet impregnated with 15 %wt. Ni. The catalysts were characterized by means of XRD, XPS, H<sub>2</sub> TPR, CO<sub>2</sub> TPD, TGA, and N<sub>2</sub> physisorption. "Post mortem" characterization of the materials was performed as well, in order to assess the impact of plasma on their structure and physico-chemical properties.

We report that the the synthesis conditions of the CeO<sub>2</sub> supports strongly influence their physico-chemical properties as well as their plasma-assisted catalytic performance. Different physico-chemical properties were considered and linked with the yield in plasma catalysis. Electrochemical characterization and in-plasma behaviour study are due in order to gain further information on the properties of these materials and to have a clearer insights on their impact on the plasma-assisted catalytic process.

## Keywords:

Plasma catalysis, non thermal plasma, dielectric barrier discharge, cerium oxide, nickel, nanostructures, CO<sub>2</sub> methanation, carbon capture and valorization



**Figure 1:** Scheme to elucidate the main points considered in this study: how the synthesis parameters affect the morphology and the physico-chemical properties of Ni/CeO<sub>2</sub> catalysts and how to match these characteristics with the catalytic activity for plasma-assisted methanation.

## References:

1. R. Dębek, F. Azzolina-Jury, A. Travert, F. Maugé, Renewable Sustainable Energy Reviews 116 (2019) 109427.

# Study on the mechanism of plasma-assisted CO<sub>2</sub> methanation over Ru-zeolite catalysts in a DBD *operando* FTIR cell.

Domenico Aceto<sup>1,2,\*</sup>, Federico Azzolina-Jury<sup>2</sup>, Carlos Henriques<sup>1</sup>

<sup>1</sup> CQE-DEQ, Instituto Superior Técnico, Universidade de Lisboa, Lisboa, Portugal

<sup>2</sup> Normandie Université, ENSICAEN, UNICAEN, CNRS, Laboratoire Catalyse et Spectrochimie, 14000, Caen, France

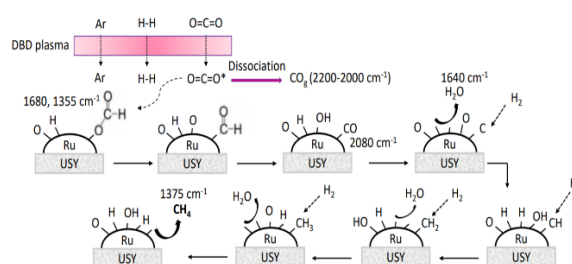
## Abstract:

CO<sub>2</sub> methanation is a well-known reaction that is advantageous in using CO<sub>2</sub> as a building block in the production of fuels, thus contributing to mitigate greenhouse gases emissions and fight the issues tied to climate change [1]. The traditional thermal pathway requires the use of a catalyst and high temperatures to activate the reaction, which could cause several drawbacks. An alternative for using thermal energy is the use of an energy source capable of activating the reactants at low temperature: non-thermal plasmas. In the last decade, in fact, a new branch of plasma science has been emerging: plasma-catalysis, whose objective is to enhance catalytic reactions by adding a plasma to the reaction cycle [2]. Of particular interest is the utilisation of dielectric barrier discharge plasma-induced catalysis, aiming at increasing products yield (by modifying energy barriers of catalytic pathway) with simultaneous low power consumptions [3]. Although promising, this novel approach poses unknown and critical aspect concerning the design of the catalyst and its interactions with plasma [4] (figure 1).

Consequently, in the present work different Ru-based zeolite supported catalysts were prepared to study their behaviour in a DBD plasma-catalytic setup, allowing to observe how different properties of the catalyst can affect the reaction pathway. A novel atmospheric pressure DBD *operando* FTIR cell was designed and utilized to study the reaction mechanism and to follow the formation of different species in the plasma and at the surface of the catalyst. CO was found to be the main product of the reaction coming from CO<sub>2</sub> dissociation by plasma in the gas phase. CO<sub>2</sub> is also vibrationally excited by the plasma, facilitating its adsorption on the surface of the catalysts in the form of oxidized carbon species (formates, carbonates, carbonyls...) that are then progressively hydrogenated to methane. Thus, a detailed mechanism for DBD-assisted CO<sub>2</sub> methanation is proposed.

**Keywords:** Ruthenium, zeolite, USY, catalyst, DBD, plasma, plasma catalysis, CO<sub>2</sub>

## methanation, reaction mechanism, *OPERANDO* FTIR spectroscopy



**Figure 1:** proposed mechanism for DBD-assisted CO<sub>2</sub> methanation over a Ru/USY catalyst, as observed through spectroscopic data obtained in a DBD *operando* FTIR cell.

## References:

1. S. Rönisch et al., Fuel 2016, 166, 276–296
2. E.C. Neyts et al., Chem. Rev. 2015, 115, 13408–13446
3. R. Debek et al., Renewable and Sustainable Energy Reviews 116, 2019, 109427
4. A. Bogaerts et al., Catalysis Today 337, 2019, 3–14



## Plasma-liquid catalysis for CO<sub>2</sub> conversion

J. Barauna<sup>1,2\*</sup>, T. M. García<sup>2</sup>, M. Magureanu<sup>3</sup>, V. I. Parvulescu<sup>1</sup>

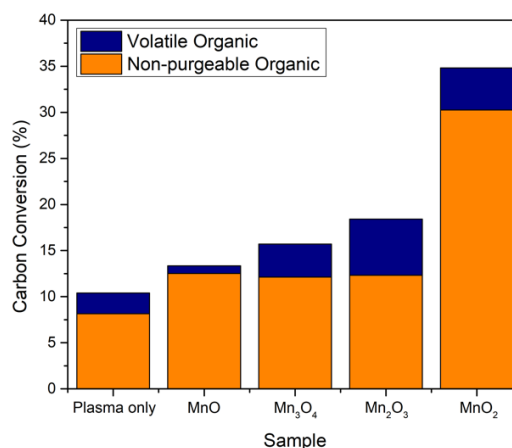
<sup>1</sup> Department of Organic Chemistry, Biochemistry and Catalysis, Faculty of Chemistry, University of Bucharest, Bucharest, Romania.

<sup>2</sup> Grupo de Investigaciones Medioambientales, Consejo Superior de Investigaciones Científicas (CSIC) – Instituto de Carboquímica, Zaragoza, Spain.

<sup>3</sup> Department for Plasma Physics and Nuclear Fusion, National Institute for Lasers, Plasma and Radiation Physics, Magurele, Romania.

**Abstract:** Plasma Catalysis has demonstrated to be a technique with extraordinary potential to convert CO<sub>2</sub> into valuable products. Indeed, synergy between plasma and catalysts is already well known to occur in gas-phase reactions, enhancing the conversion, product yield, and energy efficiency of the process as well as improving the catalyst stability by reducing poisoning, coking, and sintering<sup>1</sup>. Against this background, it should be commented that there is still a gap in knowledge concerning Plasma-Liquid interactions and the possible synergy that a catalyst can bring in such a system in the specific case of CO<sub>2</sub> conversion. Specially interesting in this field, it is the reaction between CO<sub>2</sub> and H<sub>2</sub>O since CO<sub>2</sub> activation seems to proceed by a different pathway when liquid water is used instead of gas. Whilst plasma-liquid systems lead to the formation of bigger molecules like organic acids, even in the absence of a catalyst<sup>2-4</sup>. Plasma-gas phase reactions usually produce little to no liquid products<sup>5</sup>. Herein, a tailor-made plasma-liquid slurry bubble column with CO<sub>2</sub> as the working gas has been used in order to study the possible synergy between liquid, plasma and catalysts. To accomplish that, a plasma bubble is ignited on the tip of a hollow needle from which CO<sub>2</sub> gas is flowing at 15 ml/min at the bottom of a cylindrical vessel, filled with 7 ml of water. Experiments were made in the absence of catalyst or with suspended particles of Mn-Oxides. Significantly, we show the promotion of liquid organic products by the reaction between CO<sub>2</sub> and liquid water. Preliminary Total Organic Carbon results show that CO<sub>2</sub> conversion and the type of organics is not only influenced by presence of the catalyst, but also potentially by the structure and oxidation state of the oxide used (Fig. 1).

**Keywords:** plasma-liquid, plasma catalysis, CO<sub>2</sub> conversion, CO<sub>2</sub> recycling.



**Figure 1:** Conversion of CO<sub>2</sub> to organic liquids based on Total Organic Carbon analysis of water samples after 15 min of CO<sub>2</sub> plasma using no catalyst (CO<sub>2</sub>) and different Mn-Oxides.

### References:

1. Michiels, R., Engelmann, Y. & Bogaerts, A. (2020) Plasma catalysis for CO<sub>2</sub> hydrogenation: unlocking new pathways toward CH<sub>3</sub>OH. *J. Phys. Chem. C* **124**, 25859–25872.
2. Rumbach, P., Xu, R. & Go, D. B. (2016) (2016) Electrochemical production of oxalate and formate from CO<sub>2</sub> by solvated electrons produced using an atmospheric-pressure plasma. *J. Electrochem. Soc.* **163**, F1157–F1161.
3. Kawasaki, M., Morita, T. & Tachibana, K. (2015) Facile Carbon Fixation to Performic Acids by Water-Sealed Dielectric Barrier Discharge. *Sci. Rep.* **5**, 1–9.
4. Yang, S. *et al.* (2021) CO<sub>2</sub> Reduction to Higher Hydrocarbons by Plasma Discharge in Carbonated Water. *ACS Energy Lett.* **6**, 3924–3930.
5. Snoeckx, R. *et al.* (2015) The Quest for Value-Added Products from Carbon Dioxide and Water in a Dielectric Barrier Discharge: A Chemical Kinetics Study. *Sci. Rep.* **10**, 409–424.

# In-situ water water purification method using plasma activated microbubbles for remote environments with limited resources

M. Kim<sup>1,\*</sup>

<sup>1</sup> Aeronautica and Astronautica Engineering Department, University of Southampton, Southampton, UK

## Abstract:

Water pollution is a major global problem at present, involving organic and inorganic contaminants such as heavy metals, pesticides and pharmaceutical compounds, as well as pathogens. Most of these contaminants are found in trace concentrations in water but they may exert their activity at low concentrations<sup>[1]</sup>, and potentially have an impact on drinking water supplies<sup>[2]</sup>. According to World Health Organisation (WHO), nearly a billion people lack access to clean drinking water and an estimated 3.4 million people, mostly children, die each year from water-related diseases such as cholera, diarrhoea, and gastrointestinal illness.

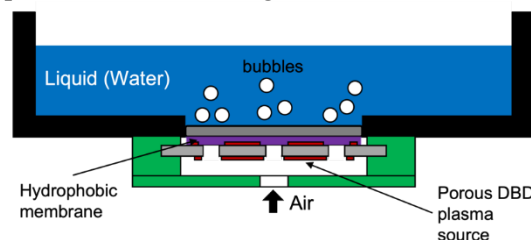
Many water-borne diseases and water-related health issues are easily preventable by improving drinking water sources. Conventional water treatment processes can achieve high removals of microorganisms and very modest removal of organic substances<sup>[3]</sup> and some inorganic compounds such as nitrate<sup>[4]</sup>. However, high amounts of toxic sludge are generated during the process, and the conventional methods are ineffective for treating pathogens and emerging contaminants such as antibiotics.

As an alternative, advanced oxidation processes (AOPs) and activated carbon adsorption can enhance the removal of organic and inorganic substances. Granular activated carbon (GAC) is known to be particularly effective in the removal of pollutants, from oil to pharmaceuticals<sup>[5]</sup>. However, GAC is expensive in terms of initial capital costs and maintenance processes associated with regeneration of the medium<sup>[6]</sup>. Hence, while its effectiveness is proven, GAC treatment cannot be adopted on large scales in developing countries. AOPs using chemical precursors need high capital and operational costs for chemical storage and handling hardware. In addition, high nitrate concentrations in the groundwater can reduce the efficiency to degrade organic compounds because nitrate would consume hydroxyl radicals, competing for hydroxyl radical reactions with the organic compounds. Therefore, it is important to develop an efficient, portable and low cost water treatment method for

contaminated drinking water for resource-limited environments.

As an alternative in-situ resource utilisation (ISRU) and recycle/reuse of wastewater, we use a non-thermal atmospheric pressure plasma to decompose chemicals and biological contaminants in water. Non-thermal plasma is a specific type of plasma that is less than 40 °C at the point of application. When non-thermal plasma contacts with water, it can efficiently generate reactive oxygen species in either gases or aqueous forms. As reactive oxygen species are key oxidants for the degradation of chemical and biological pollutants, non-thermal plasma can be used to attack and ultimately mineralise contaminants in water. As chemical reactions involving reactive species terminate in a short time, the proposing method can rapidly remove chemical and biological pollutants with no lingering residue or harmful by-products.

Although the potential of non-thermal plasma as a wastewater method has been demonstrated, little progress has been made because it requires to improve reactive species delivery. In this study, we use plasma-activated air microbubbles to overcome this technical limitation by maximising contact surface area and enhancing reactive species delivery. As can be seen in Figure 1, the developed plasma-activated air microbubble generator consists of a flexible plasma source and membrane. The implemented membrane of the proposed method keeps the electrodes on a flexible plasma source from direct contact with water and generates microbubbles to increase contact surface area between reactive oxygen plasma species and chemical agents.



**Figure 1.** A schematic of a plasma activated-microbubble water reactor.

In this study, the performance of the proposed reactor in wastewater treatment has been evaluated with methylene blue (MB) solution as the model

wastewater. We have investigated the effects of airflow rate, initial solution concentration, and plasma dose on the MB degradation efficiency. Figure 2 shows the picture of the treated water samples using a plasma activated-microbubble. As can be seen, 98% of MB has been removed after 20 minutes of plasma activated microbubble treatment. As the induced chemistry can be modified by tailoring the gas composition in the bubbles, we can optimise and control the performance of the chemical and biological contaminant removing process. Therefore, the proposed method can be a practical option for in-situ resource utilisation (ISRU) and recycling/reuse of wastewater where has limited infrastructures for water treatment.



**Figure 2.** Pictures of treated water samples using a plasma activated microbubbles, where methylene blue (MB) has been used as a simulant of chemical contaminants.

**Keywords:** water treatment, wastewater, non-thermal plasma, microbubble, decontamination, water purification.

#### References:

1. E. Vulliet and C. Cren-Olivé, *Environ Pollut*, 159(10), 2011
2. O. A. Jones, J. N. Lester, and N. Voulvoulis, *Trends Biotechnol*, 23(4), 2005
3. Rigobello et al., *Chemosphere*, 92 (2), 2013, pp. 184-191
4. Follett and Hatfield, *Nitrogen in the Environment*, 2nd Ed, 2008
5. G. Liu et al., *Journal of Hazardous Materials*, 164(2), 2009
6. R. Chowdhury et al., *Eur Heart J.*, 34(38), 2013

## Kinetic mechanisms in CO<sub>2</sub>-N<sub>2</sub> plasmas

C. Fromentin<sup>1</sup>, T. Silva<sup>1</sup>, T. C. Dias<sup>1</sup>, E. Baratte<sup>2</sup>, O. Guaitella<sup>2</sup>, O. Biondo<sup>3</sup> and V. Guerra<sup>1</sup>

<sup>1</sup> Instituto de Plasmas e Fusão Nuclear, Instituto Superior Técnico, Universidade de Lisboa, Portugal

<sup>2</sup> Laboratoire de Physique des Plasmas (UMR 7648), CNRS, Univ. Paris Saclay, Sorbonne Université, École Polytechnique, France

<sup>3</sup> Plasma Lab for Applications in Sustainability and Medicine – ANTwerp, Belgium

### Abstract:

Investigating the impact of N<sub>2</sub> on the overall CO<sub>2</sub> conversion is relevant as N<sub>2</sub> can be present as an impurity in industrial CO<sub>2</sub> emission and can be used to promote CO<sub>2</sub> vibrational excitation and further molecular dissociation through the so-called ladder climbing mechanism. The system of election is a DC glow discharge, operating at pressures in the range  $p=0.1$ -10 Torr and discharge currents  $I=10$ -50 mA, in a Pyrex tube of radius  $R=1$  cm, which is stable, axially homogeneous, and easily accessible to a variety of diagnostics. The set of measurements provides the gas temperature, vibrational temperatures of CO<sub>2</sub>, reduced field  $E/N$ , and densities of O(<sup>3</sup>P), NO, NO<sub>2</sub>, CO(X<sup>1</sup>Σ<sup>+</sup>) and CO<sub>2</sub>(X<sup>1</sup>Σ<sup>+</sup><sub>g</sub>). Our simulation results are obtained with the LoKI (LisbOn Kinetics) simulation tool [1] solving a Boltzmann-chemistry 0D self-consistent kinetic model. The comparison of the model predictions with the experimental data allows the development of a new reaction mechanism (i.e., a set of reactions and rate coefficients validated against benchmark experiments) for CO<sub>2</sub>-N<sub>2</sub> plasmas and provides physical insights into the main mechanisms occurring in these plasmas.

It is shown that the admixture of N<sub>2</sub> has a beneficial impact on CO<sub>2</sub> decomposition, as also pointed out in [2,3]. Several reasons can be assigned to it, one of them being the transfer of vibration quanta from the first vibrational level of N<sub>2</sub> to the asymmetric mode of CO<sub>2</sub> and the fact that vibrationally excited CO<sub>2</sub> can undergo molecular dissociation through the so-called ladder climbing mechanism or by electron impact step-wise processes. Moreover, the dilution with N<sub>2</sub> can also limit the influence of back reaction mechanisms producing back CO<sub>2</sub> from CO. These mechanisms will be discussed in the detail at the conference. Understanding the impact of the different processes on the overall kinetics, along with the validation against experimental data, will contribute to further develop the existing models [3-5] and to better control and enhance plasma-assisted CO<sub>2</sub> conversion.

**Keywords:** glow discharge, CO<sub>2</sub> decomposition, vibrational excitation, 0D model.

**Acknowledgments:** This work was partially supported by the European Union's Horizon 2020 research and innovation programme under grant agreement MSCA ITN 813393, and by Portuguese FCT-Fundação para a Ciência e a Tecnologia, under projects UIDB/50010/2020 and UIDP/50010/2020.

### References:

1. A. Tejero-del-Caz et al, Plasma Sources Sci. Technol. 28 (2019) 073001 [<https://nprime.tecnico.ulisboa.pt/loki>]
2. M. Grofulović et al, Plasma Sources Sci. Technol. 28 (2019) 045014
3. L. Terraz et al, J. Phys. D: Appl. Phys. 53 (2020) 094002
4. A. F. Silva et al, Plasma Sources Sci. Technol. 29 125020 (2020)
5. S. Heijkens et al, J. Phys. Chem. C 119 (2015) 12815–12828

**SICT 2022 / Tribology 2022**  
**Session III:**  
**Lubricants and hydrodynamic**  
**lubrication**

# Green Tribology: an appraisal of current status and future needs

M. Kalin

Laboratory for tribology and interface nanotechnology, University of Ljubljana, Slovenia

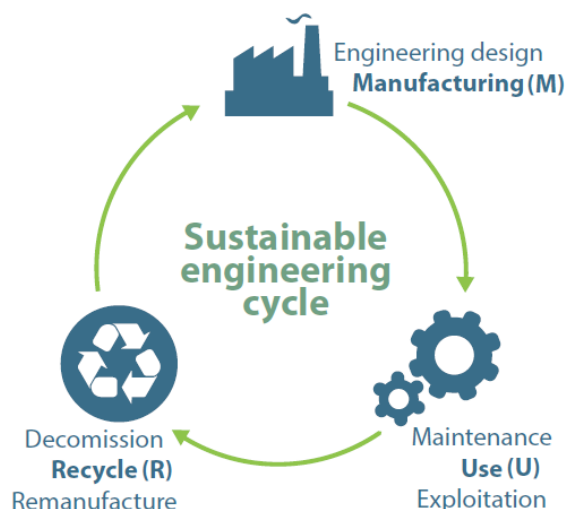
## Abstract:

Tribology has a very long history and is one of our most fundamental sciences. The Ancient Egyptians used simple lubricants, like water, to reduce the energy needed to slide huge stones and statues [1]. Modern tribology is infinitely more sophisticated, but faces the same three basic challenges: How can we reduce friction? How can we reduce wear? How can we improve lubrication? But now there is also the fourth challenge: How can we sustainably do this, by minimizing pollution, reducing the drain on natural resources, and conserving energy? We need to develop »Green Tribology« concepts to achieve this [2]. However, today we have no clear idea what truly defines green tribology, why and when this claim is justified and primarily, we do not have knowledge on tribology solutions »from the shelf« to be undisputably green and available for various applications.

Many of the activities on green sustainability are today focused on mobility applications and there indeed exists clear evidence that friction is reduced in electric vehicles [3]. However, wear should also be considered in this equation, which directly affects the green nature of the vehicles, since the long-term performance and endurance will define the need for replacements and maintenance, crucial for sustainable green engineering. However, we notice that there is much more discussion and activities to reduce friction, rather than wear. Moreover, still, very few considerations are about life-cycle analyses in tribological contacts and tribological applications, which include all phases of application lifetime, Figure 1.

In this presentation, we discuss the above issues, defining the green tribology, the current state of the art, the major needs, the impact of tribology for electric mobility, and how to obtain the appropriate knowledge to build generalized green tribology concepts.

**Keywords:** green tribology, sustainability, friction, wear, electric mobility, tribos.



**Figure 1:** Sustainable green engineering cycle for Green tribology.

## References:

1. D. Dowson, History of tribology, 2<sup>nd</sup> ed. Wiley (1998).
2. M. Kalin, et al, Green tribology for the sustainable engineering of the future, Strojnikski vestnik, Nr. 11-12 (2019) 709-727.
3. K. Holmberg, A. Erdemir, The impact of tribology on energy use and CO<sub>2</sub> emission globally and in the combustion engine and electric cars, Tribol. Int., 135 (2019) 389-396.



# The Active Role of Lubricating Grease as a Tribological System- An Energetic Approach

E.Kuhn

Department of Mechanical Engineering and Production, Hamburg University of Appl. Sc., Hamburg,

## Abstract:

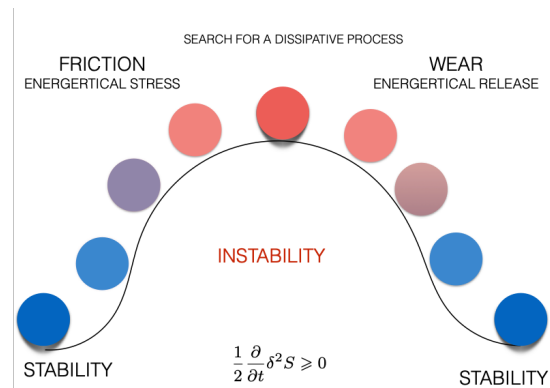
Tribology deals with defined systems which are stressed by a friction process. As an observer of that process, tribologists can record the reaction of the system in different ways and can try to interpret the obtained process behaviour. It may be interesting to look at the energetic situation and follow the course of the energetic state, because it seems to be important to determine the driving forces of the observed process [1](see Fig.1). In this study, the lubricating grease is considered as a tribological subsystem that is stressed in a contact by the friction process. A typical reaction of the lubricant is the change of the grease structure in a non-stationary-process phase. We interpret this system response as an effort by the system to eliminate the induced instability and to achieve a structure that allows a steady state.

The irreversible character of the tribological process can be observed by examining the entropy transport or entropy production [2]. The influence of the entropy transport on the strength of the system was examined using the following equation[3]

$$e_{Rrheo}^* = T_f \cdot (\rho_{out} \cdot s_{out}) - \frac{T_f}{V_{out}} (\dot{m}_{in} s_{in} - \dot{S}_Q)$$

In this lecture, the driving forces of the process when a lubricating grease is subjected to frictional stress are shown and the inherent reaction of the system is highlighted. The assumption that newly created structures in the lubricating grease also contribute the the stability of the process is given an analytical interpretation.

**Keywords:** tribology, lubricating greases, structural degradation, lubricant wear, energetic approach, entropy



**Figure 1:** Figure illustrating the effort of a tribological system to reach stability. WE track the loss of stability through the onset of a frictional process and the system's response to eliminate this disturbance [1]

## References:

1. Kuhn,E. (2020), Aspects of self-organization of tribology stressed lubricating greases, *Lubricants*,8,28,
2. Rezasoltani,A.; Khonsari,M.M, (2014) On the correlation between mechanical degradation of lubricating greases and entropy. *Tribol. Lett.* 56
3. Kuhn,E.(2015) Correlation between system entropy and structural changes in lubricating greases. *Lubricants*,3

# Development of tribological testing procedure for lubricating oil used in weaving machines.

E. Colombo<sup>1</sup>, M. Toscanini<sup>1</sup>

<sup>1</sup> R&D downstream research center, Eni SpA, Via Felice Maritano 26, 20097 San Donato Milanese, Italy

## Abstract:

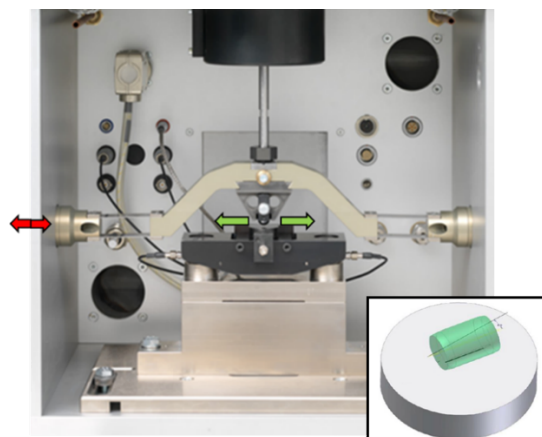
The textile industry exploits different kinds of spinning, knitting, and weaving machines; each of them needs proper lubrication. In particular, mechanical sliding parts in weaving machines are subjected to oscillation, vibrations, high loads, and speeds; therefore, lubricating oils have to provide wear protection and scuffing load resistance in addition to outstanding anti-oxidation properties, since oil-change intervals may reach 20'000 hours. The scope of the developed experimental activity is to outline an ad hoc tribological test for relative motion able to discriminate oils with promising lubrication properties against seizure and stick-slip from unsuitable lubricants. The test focuses on the evaluation of friction and wear using SRV®5 apparatus developed by Optimol. The tribological coupling, which is closer to real tribological contact, is cylinder-on-disk (Figure 1). The cylinder, made of steel, is forced to move in reciprocating oscillatory motion against the disk made of a special alloy. Tight collaboration with textile machine manufacturer is of pivotal importance in order to tune test parameters, i.e. load and temperature, simulating real operating conditions within weaving machines. Friction coefficient (COF) is recorded throughout the test duration of 8 hours; wear scar characteristics in terms of depth and width are evaluated at the end of the test using a profilometer. Two reference lubricants are chosen: a commercial oil, commonly used in textile gearboxes, and the first formulated product, which led to seizure of mechanical parts in previous bench test. Different lubricants are formulated with low viscosity synthetic base oils and specific additives. The best candidate oil presents excellent performance and promising energy-saving properties. Results of the tribological test have to be confirmed with real scale test scheduled for the next year.

In order to support product design, standard tribological and physico-chemical tests are performed. Anti-wear and EP properties are confirmed through 4-ball wear and EP. Stick-slip tests are carried out to verify stick-slip ratio that has to be lower than one. Besides, foaming tendency needs careful evaluation since foam must

be avoided within the lubricating circuit. Washability, safety features and anti-corrosion properties are checked too.

In conclusion, this experimental activity leads to the definition of an innovative in-house tribological test, which helps oil formulators in differentiating the lubricating ability of oils in weaving machines according to friction and wear results. Finally, this methodology helps to gain deeper insight into the lubrication mechanisms for textile machines.

**Keywords:** tribology, SRV®5, weaving machines, energy-saving lubricant.



**Figure 1:** SRV®5 test system and tribological coupling cylinder-on-disk.

## References:

1. Mang, T. (2014) Encyclopedia of Lubricants and Lubrication, *Springer*, 2081-2083.
2. Patzer, G., Ebrecht, J. (2016) Vorstellung eines Pruefkonzepts als Screening-methode für Getriebeoole auf dem translatio-rischen Oszillationstribometer (SRV®), *Tribologie und Schmierungstechnik*, 63, 2, 58-65 (German).

# Tribological Performance of Lubricating Greases Composed by Different Bio-based Polymer Thickeners under EHL Conditions

S. Vafaei <sup>1,\*</sup>, G. Jacobs <sup>1</sup>, F. König <sup>1</sup>, R. Weberskirch <sup>2</sup>

<sup>1</sup> Institute for Machine Elements and System Engineering (MSE), RWTH Aachen University, Aachen, Germany

<sup>2</sup> Faculty of Chemistry and Chemical Biology, Technical University of Dortmund, Dortmund, Germany

## Abstract:

The lubricant industry develops and produces lubricating oils and greases for the lubrication of machine elements such as rolling element bearings. Lubricants are essential to reduce friction and thus energy consumption on the one hand, and to prevent a premature failure of machine elements due to wear or fatigue. Most lubricants available on the market today are still produced petrochemically, i.e., on the basis of fossil raw materials. The importance of substituting fossil-based materials with renewable materials has been discussed recently across all industry sectors. This has also motivated the lubricant industry to apply sustainable lubricants. Normally a bearing grease is based on around 3 to 30 wt.% thickener and the rest is base oil as well as a small amount of additive (approx. 0-10 wt.%). There are some bio-based lubricants available on market, which are usually ester based oils. However, to produce an entirely bio-based grease, the thickener must also be produced from renewable materials.

This work presents the extended design and evaluation of different developed bio-based polymer thickener systems. Tribological tests are performed to characterize lubrication properties of developed bio-based greases under EHL conditions. The effect of thickener type on film thickness and friction behaviour of the produced bio-based greases is evaluated on an EHD2 ball-on-disc tribometer (PCS Instruments). Moreover, the results are compared to a commercial petrochemical grease chosen as benchmark. The bio-based alternatives show a similar performance to the petrochemical-based greases. Further steps include the wear and fatigue performance analysis on component test rigs for rolling element bearings.

**Keywords:** Biodegradability, bio-based grease; grease lubrication; film thickness; friction measurement; polymer thickener; ball-on-disc tribometer.

# Development of sustainable and effective water-based metalworking fluid for titanium machining

E. Benedicto <sup>1,2,\*</sup>, E.M. Rubio <sup>1</sup>, L. Aubouy <sup>2</sup>, M.M. Marín <sup>1</sup>

<sup>1</sup> Department of Manufacturing Engineering, Industrial Engineering School, Universidad Nacional de Educación a Distancia (UNED), Madrid, Spain

<sup>2</sup> Department of Surface Chemistry, Leitat Technological Center, Terrassa, Spain

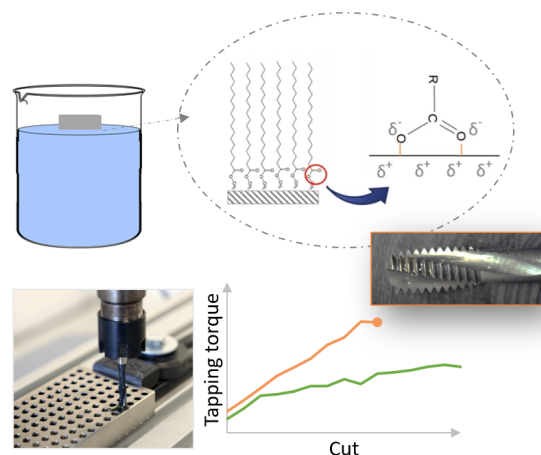
\*Programa de Doctorado en Tecnologías Industriales

## Abstract:

Metalworking fluids (MWF) can increase the productivity, sustainability, and quality of machining processes, especially for difficult-to-cut materials, such as titanium alloys, where most of the problems are related to the high consumption of cutting tools due to excessive wear. Cutting fluids are used to improve the machining process by providing lubrication, dissipating heat, and removing chips from the cutting zone. However, MWFs are under review due to their environmental impact and health risks to workers. There is an urgent need for the development of new sustainable MWFs, and it still represents a demanding challenge.

Herein, we study the influence of surfactant and esters' molecular structure in oil-in-water emulsions and its interaction with the metal surface to form a lubricating layer, thus improving the performance of the MWF. Lubrication and tool wear protection are studied through film formation analysis and the tapping process on Ti6Al4V (Figure 1). By modifying the molecular structure of the surfactant, it is possible to enhance the affinity between the ester and the substrate and reach an optimal mixture, which enhances the formation of a tribofilm [1]. Polyol esters show promising results to replace mineral oils. Specifically, a higher lubricating film is formed with trimethylolpropane trioleate, thus improving the lubricity by up to 12% and reducing tool wear by 26.8% [2]. The applied technology of this work may be helpful for the development of new environmentally friendly MWF, not only for titanium machining but also for the design of MWF for conventional and advanced alloys.

**Keywords:** metalworking fluid, cutting fluid, surfactant, ester, lubrication, tool wear, tool life, difficult-to-cut materials, titanium alloys, Ti6Al4V.



**Figure 1:** Test set-up diagram of the analysis of tribofilm and the tool wear during tapping to study the role of surfactants and esters on the development of sustainable and effective MWF.

## References:

1. Benedicto E, Rubio EM, Carou D, Santacruz C. The Role of Surfactant Structure on the Development of a Sustainable and Effective Cutting Fluid for Machining Titanium Alloys. *Metals* (Basel) 2020;10:1388. doi:10.3390/met10101388.
2. Benedicto E, Rubio EM, Aubouy L, Sáenz-Nuño MA. Formulation of Sustainable Water-Based Cutting Fluids with Polyol Esters for Machining Titanium Alloys. *Metals* (Basel) 2021;11:773. doi:10.3390/met11050773.

## Development of HPDC system for lubricants evaluation

L. Batlle<sup>1</sup>, E. Benedicto<sup>1</sup>, D. Cecilia<sup>2</sup>, L. Muntada<sup>3</sup>, W. Ajana<sup>3</sup>

<sup>1</sup> Department of Surface Chemistry, Leitat Technological Center, Terrassa, Spain

<sup>2</sup> Department of Technology Integration & Product Development, Leitat Technological Center, Terrassa, Spain.

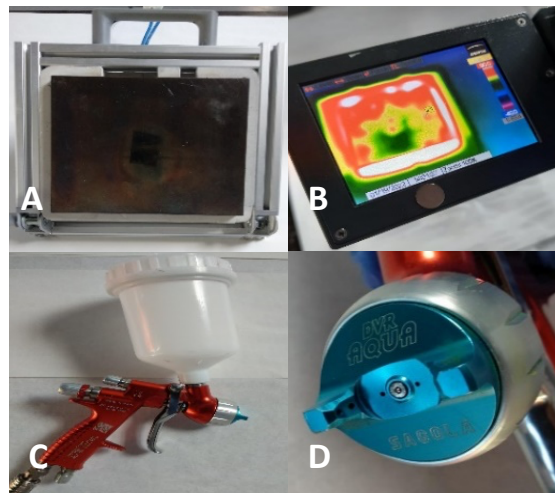
<sup>3</sup> Brugarolas S.A, Rubí, Spain.

### Abstract:

Lubricants are commonly used in High-Pressure Die Casting (HPDC) processes to cool, protect parts from corrosion, prevent welding effects and improve the removal of finished products. These lubricants are sprayed all over the surface before the liquid metal is introduced under pressure into die cavities. However, the costs related to product development are high due to the lack of representative systems at the laboratory scale. There is an urgent need to develop a lab-scale methodology that contributes to understanding spray mechanism, die surface temperature distribution after lubricant application, and the Leidenfrost point effect [1]. In this research, a new lab system has been developed to simulate the cooling effect of the mold (Figure 1). The aluminum structure is designed to modify the surface angle position, allowing to change the spray angle of incidence. Test die is manufactured of steel H13 placed on a ceramic support. The equipment includes a 500W resistance distributed along the test die to heat it over the working temperatures. Generally, in the die casting process, the mold reaches an average temperature of 200-300°C [2]. In addition, three thermocouple sensors and a heat flux sensor register the surface temperature at different surface points.

To study the performance of several mold release agents, first, the mold is heated at the desired temperature. Then, the HPDC lubricant is applied to the mold with a spray gun at a fixed distance from the surface. During the spraying stage, the cooling effect of the mold release agents is evaluated using a thermographic camera. Moreover, the Leidenfrost effect (droplets reaching hot's surface) is observed with a slow mode camera (240 fps, 4K). Promising outcomes on the spraying mechanism of each lubricant have been obtained. The described system may be useful for developing more effective HPDC lubricants, allowing to evaluate their efficiency.

**Keywords:** high pressure die casting, HPDC, lubricant, mold release agent, lab scale methodology, Leidenfrost point.



**Figure 1:** Set-up system including steel mold (A), thermographic camera (B), spray gun (C), and spray nozzle (D).

### Acknowledgments:

The authors wish to acknowledge the financial support received from the Spanish Ministry of Economy and Competitiveness; within the strategic Business Research Consortium Program (CIEN) 2019, from the Center of Industrial Technological Development (CDTI). The project with the acronym LUBRINTEL has been developed under the guidance and with close collaboration between Brugarolas SA, Eurecat Technological Center and Leitat Technological Center.

### References:

- [1] Y. Kouki, S. Müller, T. Schuchardt, and K. Dilger, 'Development of an Instrumented Test Tool for the Determination of Heat Transfer Coefficients for Die Casting Applications', *Metals*, vol. 10, no. 9, p. 1206, Sep. 2020, doi: 10.3390/met10091206.
- [2] A. Long, D. Thornhill, C. Armstrong, and D. Watson, 'Predicting die life from die temperature for high pressure dies casting aluminium alloy', *Applied Thermal Engineering*, vol. 44, pp. 100–107, Nov. 2012, doi: 10.1016/j.applthermaleng.2012.03.045.



# Study of friction reducers obtained from Sargassum algae: Effect of the structure on the tribological performances of the carbon phases

A.Molza<sup>1</sup>, P.Bilas<sup>1</sup>, T.Cesaire<sup>1</sup>, P.Thomas<sup>1</sup>, Y.Bercion<sup>2</sup>

<sup>1</sup>Groupe de Technologie des Surfaces et Interfaces (GTSI EA2432), Université des Antilles et de la Guyane, Pointe à Pitre, Guadeloupe (France)

<sup>2</sup>Centre Commun de Caractérisation des Matériaux des Antilles et de la Guyane (C<sub>3</sub>MAG), Université des Antilles et de la Guyane, Pointe à Pitre, Guadeloupe (France)

## Abstract:

As it is well known, conventional lubricant additives used to reduce wear and friction in boundary lubrication regime act by formation of protective tribofilms on the rubbing metallic surfaces by reaction, in the physico-chemical conditions of their sliding interface [1,2]. The efficiency of such molecular additives depends on the reactivity of the surfaces and consequently on the kinetic of the tribofilm built up.

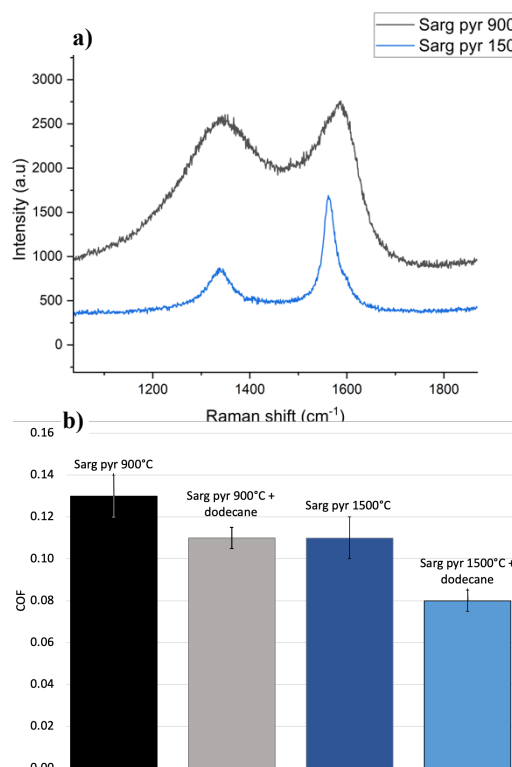
In order to overcome the limitations of such anti wear and friction reduction additives used in lubrication oils and greases, new strategies, using solid nano particles as nano additives, were recently developed [3, 4].

In Caribbean islands, washing ashore of tons of pelagic Sargassum *spp.* consisting of two species Sargassum *fluitans* and Sargassum *natans* have been regularly occurring since 2011.

The present work is concerned with the tribological properties of the carbon phase resulting from pyrolysis of Sargassum algae under different pyrolyzed temperature (900°C and 1500°C) used as solid lubricant and dispersed in the lubricating base oil presenting a low viscosity (dodecane). As for molecular additives, the friction reduction process is associated to the formation of a tribofilm between the rubbing surfaces [3,4]. The effect of the structure of carbon phase (fig 1 - 2) on the friction reduction performances is studied.

The morphology and the structure of the tribofilm are investigated by profilometry, scanning electron microscopy and by Raman microspectrometry.

**Keywords:** tribology, carbon material, algae biomass, carbonization, Raman spectroscopy



**Figure 1:**

**a)** Raman spectrum recording on initial carbon phase resulting from pyrolysis of Sargassum alage under 900°C and 1500°C. Raman spectrum revealed that both carbon phases present two dominant peaks at around 1347 cm<sup>-1</sup> (D-band) and 1596 cm<sup>-1</sup> (G-band). The ratio between ( $I_D/I_G$ ) was usually used to evaluate the graphitization degree of the carbon structure. The calculated integrated intensity of  $I_D/I_G$  for Sarg 900°C and Sarg 1500°C is separately about 4.2 and 1.4.

**b)** Friction coefficient values obtained in dry and in the presence of dodecane on carbon phase resulting from pyrolysis of Sargassum alage under 900°C and 1500°C.

## References:

1. Martin, J.M., Mansot, J.L., Berbezier, I., Belin, M., "Microstructural aspects of lubricated mild wear with zinc dithiophosphate", Wear, 107, 1986, 355-366.
2. Spikes, H., "The history and mechanisms of ZDDP". Tribol. Lett., 17, 3, 2004, 469-489,



3. Mansot, J.L., Hallouis, M., Martin, J.M.” Colloidal antiwear additives. Part two: Tribological behaviour of colloidal additives in mild wear regime”, Colloids and Surfaces A, 75, 1993, 25-31.
4. Mansot J.L., Martin J.M., Bercion Y., Romana L., “Nanolubrication”, Brazil. J. of Phys., 39, 1, 2009,

# Mechanism analysis of tribological performance enhancement using MoS<sub>2</sub> and h-BN nanomaterials as nano-additives into poly alpha olefin

Hua Jiang <sup>a,b</sup>, Xianjun Hou <sup>a,\*</sup>, Mohamed Kamal Ahmed Ali <sup>c</sup>, Karl D. Dearn <sup>b,\*\*</sup>

<sup>a</sup> Hubei Key Laboratory of Advanced Technology for Automotive Components, Wuhan University of Technology, Wuhan 430070, China.

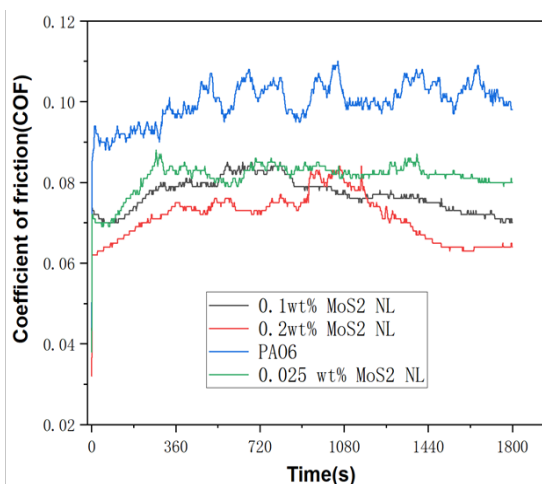
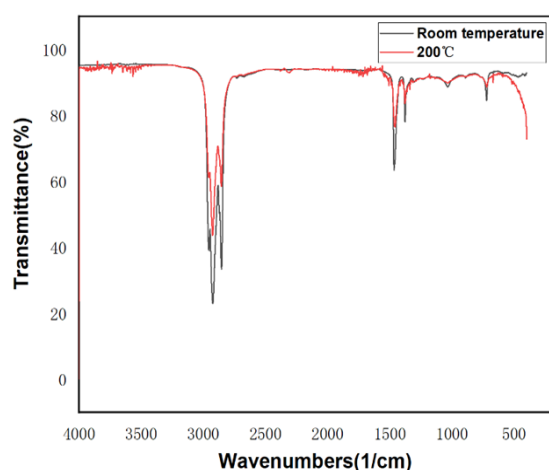
<sup>b</sup> Mason Institute of Tribology, Department of Mechanical Engineering, School of Engineering, University of Birmingham, Edgbaston, Birmingham B15 2TT, UK

<sup>c</sup> Automotive and Tractors Engineering Department, Faculty of Engineering, Minia University, El-Minia 61519, Egypt

## Abstract

Friction and wear are the severest parts of automobile engine power loss, which has an important impact on vehicle emissions, power performance, and fuel economy. Excellent lubricants are essential for an improvement of friction performance between engine friction pairs. The tribological performance of steel ball lubricated by polyalphaolefin (PAO 6) oil introducing MoS<sub>2</sub> and h-BN nanoparticles functionalized by oleic acid (OA) at different concentrations was investigated. In this research, h-BN and MoS<sub>2</sub> nanoparticles possessing diverse concentrations (0.025-0.2wt%) were evenly and stably dispersed in poly alpha olefin (PAO6). The rheological properties of each nano lubricant are introduced by

measuring the change of viscosity with shear rates at a certain temperature. Reducing-friction and anti-wear peculiarities of each nano lubricants (NL) were comprehensively assessed. Furthermore, the surface morphology, element component, and distribution were detected and characterized by field emission scanning electron microscopy (FESEM), Energy disperse spectroscopy (EDS), and XPS. Eventually, the tribological results showed that the coefficient of friction of wear surface lubricated by h-BN and MoS<sub>2</sub> NL was greatly decreased compared with PAO6 oil. Meanwhile, a feasible and convincing lubricating mechanism by nano-additives was proposed.



# Tribological response of self-mated Zircaloy-4 under dry and water submerged conditions

Bharat Kumar<sup>1</sup>, Deepak Kumar<sup>1\*</sup> and Vijay Chaudhry<sup>2</sup>

<sup>1</sup>Center for Automotive Research and Tribology (CART), Indian Institute of Technology Delhi, New Delhi, India

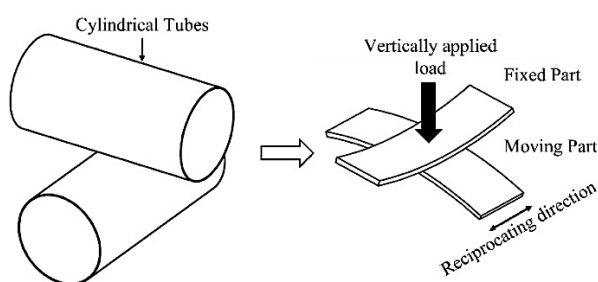
<sup>2</sup>Nuclear Power Corporation of India Limited, Mumbai, Maharashtra, India

\*Corresponding author: [dkumar@iitd.ac.in](mailto:dkumar@iitd.ac.in)

## Abstract:

Zirconium alloys are excellent materials having low neutron absorption cross-section<sup>1</sup> and good mechanical properties<sup>2</sup>, due to which these are widely used for core components in nuclear reactors<sup>3</sup>. Reciprocating tribo-tests were conducted on Zircaloy-4 (Zr-4) under the self-mated conditions (Fig. 1) at varying amplitude, frequency, and test duration. To understand the active wear mechanism, worn surfaces were analyzed using Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), Raman spectroscopy and 3D Profilometry. The change in amplitude severely affects the wear than other factors. The wear mechanism transits from stick-slip to gross slip with increasing amplitude. The dominant wear mechanisms were delamination at the amplitude of 100  $\mu\text{m}$  whereas micro-cutting and micro-ploughing followed by delamination in some areas at the amplitude of 400  $\mu\text{m}$ . However, the COF has indifferent behaviors under dry conditions, whereas it remains almost constant for the given parameters under water submerged conditions. Generally, COF and wear were less for water submerged conditions than for dry conditions. The oxide layer was also found on worn surfaces, which is prominent at 100  $\mu\text{m}$  amplitude and decreases with increasing amplitude (present only at a few locations at 400  $\mu\text{m}$  amplitude).

**Keywords:** Zircaloy, Fretting wear, adhesion, delamination, micro ploughing.



**Fig. 1:** schematic diagram of tribo-pair

## References:

1. A. Ichihara, S. Kunieda, and K. Shibata, (2009) Calculation of Neutron Cross Sections on 90; 91; 92; 94; 96Zr for JENDL-4, *J. Nucl. Sci. Technol.*
2. N. Khare, P. K. Limaye, K. Singh, D. T. Jadhav, A. Bute, and N. Kalel, (2018) Experimental and Theoretical Analysis of Zircaloy-4 and AISI 304 Stainless Steel Material Pair in Water Sliding Conditions, *Adv. Tribol.*
3. F. Onimus, S. Doriot, and J.-L. Béchade, (2020) 3.01 - Radiation Effects in Zirconium Alloys, R. J. M. Konings and R. E. B. T.-C. N. M. (Second E. Stoller, Eds). *Oxford: Elsevier.*

# Performance of non-toxic, corrosion resistance, and lubricious metal-working fluid under machining

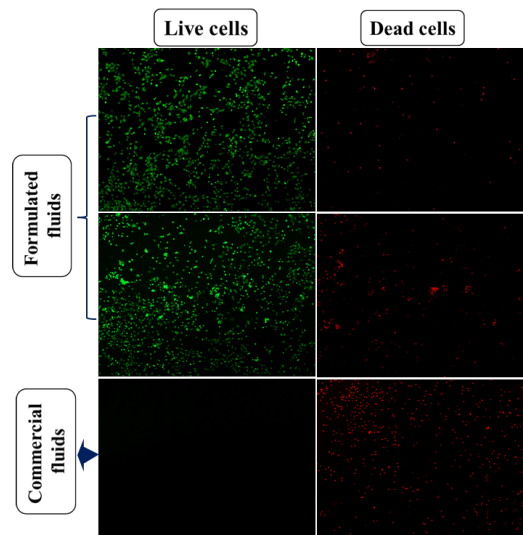
Ajay Pratap Singh Lodhi <sup>1\*</sup>, Deepak Kumar <sup>1</sup>

<sup>1</sup> Centre for Automotive Research and Tribology, Indian Institute of Technology, Delhi, India

## Abstract:

Petroleum, and mineral oil-based metalworking fluids (MWFs) are widely used in machining operations. Mineral oil-based MWFs are unsafe to humans and the environment because they comprise toxic and harmful chemicals. The harmful effects accompanying mineral oil-based MWFs can be reduced by environmentally friendly MWFs. Therefore, in the present study environmentally friendly vegetable oil-based water-soluble metalworking fluids (MWFs) are formulated. The stability, droplet size and distribution, and wettability study of the formulated MWFs were carried out on Zetasizer, Dynamic Light Scattering, and Goniometer, respectively. The cytotoxicity, corrosion, and the tribological performance of the formulated fluids was evaluated benchmarked with the commercial mineral oil-based water-soluble MWFs. Tribological testing of formulated MWFs was carried out on under machining operation. Results showed that formulated fluids exhibited better machining characteristics (surface roughness, cutting forces, and surface morphology) compared to the commercial MWF. MTT assay and Live dead cell assay confirm the cytocompatibility nature of the formulated fluids relative to the toxic commercial MWF (Figure 1). An electrochemical study showed that formulated MWFs and commercial MWFs showed comparable corrosion current density. The improved performance of the formulated MWFs is attributed to their film-forming ability at the tribo-pair interface. The chemisorption of the emulsifier molecules on the machined steel surface leads the lubricious Tribofilm formation. The nature and presence of the lubricious film on the machined steel surface were confirmed by SEM-EDS mapping and FTIR spectroscopy analysis.

**Keywords:** Cytotoxicity, Corrosion inhibitors, Vegetable oil, MTT assay, Machining, Tribofilm



**Figure 1:** Live and Dead cell assay of the formulated and commercial MWFs

# Virtual Presentations

# **SICT 2022 / Plasma Tech 2022 joint virtual session**



## Biobased functional coatings for cellulosic substrates

M.B.Coltelli<sup>1,2</sup>, L.Panariello<sup>1,2</sup>, V.Gigante<sup>1,2</sup>, L. Aliotta<sup>1,2</sup>, S. Giangrandi<sup>3</sup>, Ahdi Harich<sup>5</sup>, I. Canesi<sup>5</sup>, A. Lazzeri<sup>1,2,5</sup>, P. Cinelli<sup>1,2</sup>

<sup>1</sup> National Interuniversity Consortium of Materials Science and Technology (INSTM), Firenze, Italy;

<sup>2</sup> Department of Civil and Industrial Engineering, University of Pisa, 56126 Pisa PI, Italy;

<sup>3</sup> LUCENSE SCA RL, Lucca, Italy;

<sup>4</sup> Biomass Valorization Platform-Materials, CELABOR s.c.r.l., Chaineux, Belgium;

<sup>5</sup> Planet Bioplastics s.r.l., Pisa, Italy

### Abstract:

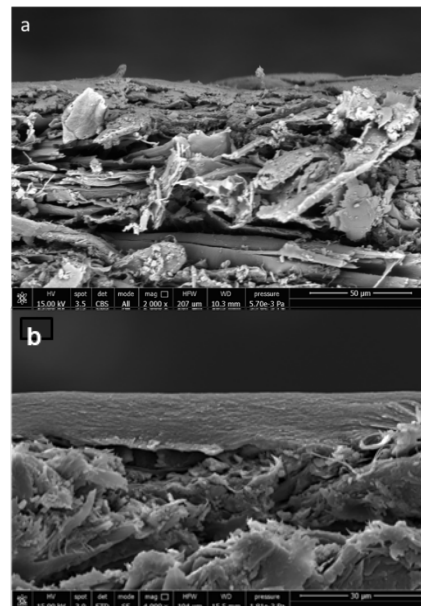
Cellulosic substrates, as renewable and recyclable items, are highly used in the packaging and personal care field. However, to expand their use and improve their properties, the application of a functional coating improving the barrier to oxygen, water vapour of the substrate is fundamental in packaging applications. Moreover, to confer anti-microbial/anti-oxidant properties to substrates addressed to personal care applications can be really useful. This coating should be biobased and not affecting the end-of life of the final packaging or personal care product (recycling and/or composting). Several coatings were proposed based on biopolymers like proteins, chitin or cutin [1].

Chitin is a very abundant polymer available from crustaceans exoskeletons (sea food waste), from insects or from mushrooms. It can be converted to chitin nanofibrils or chitosan on the basis of the adopted process. Chitin nanofibrils (CN) and chitosan based coatings can confer to cellulosic board anti-microbial properties, so they can be considered as functional coatings for increasing the shelf life of packaging for fresh food [2], but also as anti-microbial/skin-regenerative additives for applications considering the contact with human skin [3].

We report the results related to the application of chitin derivatives to several cellulosic substrates with different thickness and mechanical properties. Cellulosic board and tissue will be compared not only in terms of the capacity of chitin nanofibrils to confer anti-microbial properties, but also considering their impact onto mechanical properties.

The application of these functional biopolymers to cellulosic substrates could occur by using both liquid or solid coatings [1], that can be prepared by peculiar polymer processing methodologies. All these possibilities could represent valid alternatives to fossil coatings in the future.

**Keywords:** biobased coatings; Functional coatings; chitin;



**Figure 1:** Micrographs of sections of cellulose board: (a) recycled board (b) CN treated recycled board

### References:

1. Gigante, V., Panariello, L., Coltelli, M.B., Danti, S., Obisesan, K.A., Hadrich, A.; Staebler, A.; Chierici, S.; Canesi, I.; Lazzeri, A. Cinelli, P. (2021), Liquid and Solid Functional Bio-Based Coatings. *Polymers* 13, 3640.
2. Panariello, L., Coltelli, M.B., Buchignani, M., Lazzeri, A (2019), Chitosan and nanostructured chitin for biobased anti-microbial treatments onto cellulose based materials, *Eur. Polym. Jour.*, 113, 328
3. Coltelli, M.B., Aliotta, L., et al. (2020), Properties and Skin Compatibility of Films Based on Poly(Lactic Acid) (PLA) Bionanocomposites Incorporating Chitin Nanofibrils (CN). *J. Funct. Biomater.* 11, 21.

**Acknowledgements:** This research was funded by the Bio-Based Industries Joint Undertaking under the European Union Horizon 2020 research program (BBI-H2020), ECOFUNCO project, grant number G.A 837863;

## Growth of carbon-based fibrous nanostructures via Chemical Vapor Deposition on half-Heusler alloys

A. Manasi<sup>1</sup>, I.G. Aviziotis<sup>1</sup>, A.F.A. Trompeta<sup>1</sup>, A. Ntziouni<sup>1</sup>, S. Deligiannis<sup>2</sup>, A. Alexandratou<sup>1</sup>, E.P. Koumoulos<sup>2</sup>, C.A. Charitidis<sup>1,\*</sup>

<sup>1</sup> Research Lab of Advanced, Composite, Nano-Materials and Nanotechnology (R-NanoLab), Materials Science and Engineering Department, School of Chemical Engineering, National Technical University of Athens, 9 Heroon Polytechniou Street, Zografos, Athens 15780, Greece

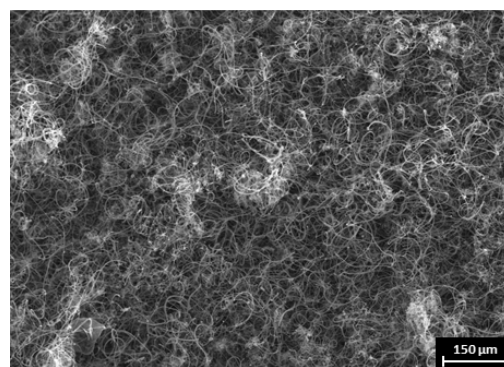
<sup>2</sup> Innovation in Research & Engineering Solutions (IRES), Rue Koningin Astridlaan 59B, 1780, Wemmel, Belgium

### Abstract:

The allotropic forms of carbon with tubular nanostructure have presented excellent physical and chemical properties and attracted the attention of scientists. The significant thermal and electrical conductivity, the high tensile strength and their flexibility, are some of the properties that can be exploited in energy storage and electrochemical devices [1]. The most promising method for producing CNTs or CNFs, is the Chemical Vapour Deposition (CVD) owing to its low-cost, relatively low applied temperature and controllable growth. The most common catalytic materials for CNTs/CNFs synthesis are Fe, Co, Ni and their alloys, mainly due to their ability to decompose carbon precursors and their mediate to high carbon solubility, yielding the carbon diffusion through their bulk and the surface formation of graphitic structures during cooling [2]. In the present work, it is investigated for the first time, the possibility of directly growing CNTs and CNFs on half-Heusler alloys by CVD, in an attempt to improve the thermoelectric performance of these compounds, by forming a conductive coating on their surface. Till now, the main investigation on half-Heusler alloys focuses on their electronic structure and their magnetic and thermoelectric properties. There are also few works investigating their potential catalytic properties with the added value being the control of the electronic structure and the change of surface elements by substitution [3]. In this work, the catalytic properties of half-Heusler compounds in a CVD process are tested. As carbon source, acetylene is used over two different half-Heusler, namely (ZrTi)Ni(SnSb) and (ZrTi)Co(SnSb), in the temperature range of 600°C - 700°C. The SEM analysis confirms the growth of CNFs with diameters ranging from 450 nm to 1 µm with respect to the operating conditions (Figure 1). The Raman modes imply the formation of carbon structures closer to CNFs rather than CNTs which co-exist with amorphous carbon. Additionally, nanoidentation technique is used to assess the uniformity of the fibrous coating and

provide information regarding its adhesion to the substrate. From the above characterization results, it may be concluded that a short reaction time and a low acetylene flow rate, lead to the formation of a uniform and stable CNF mat on the specimens, while Ni favours the reaction. Further investigation is required for process optimization for CNT growth.

**Keywords:** Heusler alloys, CNTs, CNFs, CVD, nanoindentation, Raman, thermoelectrics.



**Figure 1:** SEM micrograph of the deposited CNFs on (ZrTi)Ni(SnSb).

**Acknowledgments:** This work was funded by the EU H2020 Project 'FAST and Nano-Enabled SMART Materials, Structures and Systems for Energy Harvesting' (FAST-SMART) under Grant no. 862289. The authors would like to acknowledge MBN nanomaterialia for the provision of the samples.

### References:

1. Paradise, M., Goswami, T. (2007) Carbon nanotubes – Production and industrial applications, *Mater. Des.*, 5, 1477-1489.
2. Liu, W.W., Chai, S-P., Mohamed, A.R., Hashim, U. (2014) Synthesis and characterization of graphene and carbon nanotubes: a review on the past and recent developments, *J. Ind. Eng. Chem.*, 20, 1171-1185.
3. Kojima, T., Kameoka, S., Tsai, A-P. (2019) The emergence of Heusler alloy catalysts, *Sci. Technol. Adv. Mater.*, 20, 445-455.

# Fabrication of worm-like carbon fibers from Oligomer-rich soot generated by Solution Plasma

Andrés E. Romero V.<sup>1</sup>, Chayanaphat Chokradjaroen<sup>1</sup>, Nagahiro Saito<sup>1,2,3,4</sup>

<sup>1</sup> Department of Chemical Systems Engineering, Graduate School of Engineering, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

<sup>2</sup> Conjoint Research Laboratory in Nagoya University, Shinshu University, Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

<sup>3</sup> Japan Science and Technology Corporation (JST), Open Innovation Platform with Enterprises, Research Institute and Academia (OPERA), Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

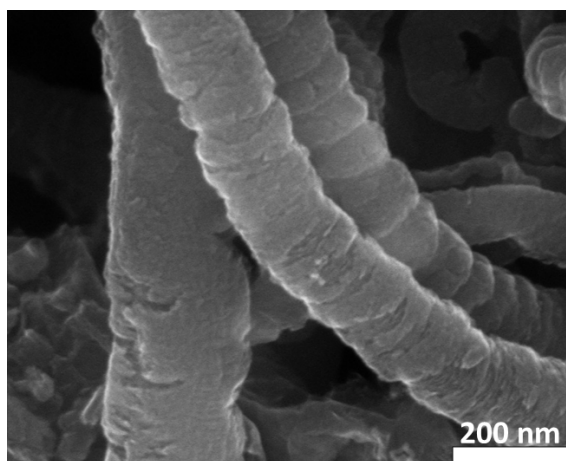
<sup>4</sup> Japan Science and Technology Corporation (JST), Strategic International Collaborative Research Program (SICORP), Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

## Abstract:

Solution Plasma (SP) is a recognized method for producing nanomaterials such as carbon sheets and heterographene [1]. When a non-equilibrium plasma is applied to organic solutions, carbon materials can be synthesized at room temperature. The ions, electrons and radicals generated in the plasma center and plasma-liquid interface can further react with the surrounding molecules leading to polymerization of carbon materials[2]. Several investigations have been reported about reactions in solution. Nevertheless, the species evaporated from the liquid phase in the form of oligomer-rich soot that is produced during the non-equilibrium plasma discharge have been rarely investigated. In the present work, the oligomers that are light enough to be carried out from the solution in the SP reactor during the plasma discharge, were further used as precursor for the fabrication of carbon fibers. The growth of fibers was similar to the mechanism reported by Endo, 1978 [3], in which the oligomer-rich soot is deposited on a substrate and the carbonaceous species grow into worm-like carbon fibers. The growth was conducted under atmospheric pressure and low temperature (400°C).

Moreover, the use of different organic solutions containing different functional groups enables the simultaneous fabrication and functionalization of the carbon fibers.

**Keywords:** Carbon fiber, solution plasma, oligomers, deposition, organic compound, functional group.



**Figure 1:** SEM image of carbon fibers fabricated by Solution Plasma (SP) generated oligomers grown on a Ni substrate.

## References:

1. S. Chae, G. Panomsuwan, M. A. Bratescu, K. Teshima and N. Saito. (2019) p-Type doping of graphene with cationic nitrogen. *ACS Appl. Nano Mater.*
2. T. Morishita, T. Ueno, G. Panomsuwan, J. Hieda, A. Yoshida, M.A. Bratescu, N. Saito. (2016) Fastest formation routes of nanocarbons in solution plasma processes. *Scientific Reports* 6, 36880.
3. M. Inagaki. (2000), New carbons – control of structure and functions. *Elsevier science*. 82-117.

# Fabrication of Anti-Wetting Coatings for Cold Environments

A. Corozzi<sup>1,\*</sup>, F. Veronesi<sup>1</sup>, J. Mora<sup>1,2</sup>, M. Caruso<sup>1</sup>, M. Raimondo<sup>1</sup>

<sup>1</sup> Institute of Science and Technology for Ceramics, National Research Council (ISTEC CNR),  
Via Granarolo, 64, Faenza, 48018 Italy

<sup>2</sup> Ingeniería de Sistemas para la Defensa de España SA, Beatriz de Bobadilla 3, Madrid, 28040 Spain

## Abstract:

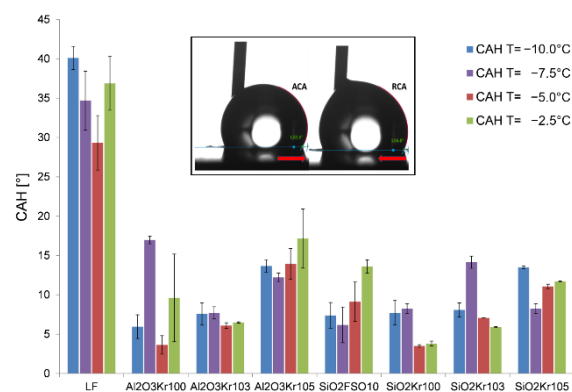
The formation and following accretion of different forms of ice poses serious safety and operational challenges in wind farms and airplanes, high voltage power lines, telecommunication systems, condenser surfaces, offshore platforms, locks and dams. Intense efforts are therefore dedicated to the development of passive ice protection systems (IPS) able to control or prevent ice formation. In particular, anti-wetting materials applied on the target surface have been explored as potential icephobic surfaces [1], with the Slippery, Liquid-Infused Porous Surfaces approach (SLIPS) being one of the most innovative and intriguing possible solutions to inhibit the ice accretion or weaken the ice adhesion strength without any power supply [2].

We present the design of anti-wetting hybrid coatings for cold environments that comprise an inorganic, porous layer based on ceramic ( $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ) nanoparticles obtained via sol-gel, while the organic layer consists of fluoroalkylsilane molecules. For SLIPS, either a fluorinated or a fluorine-free oil was infused in the porous hybrid coatings [3]. Through Icing Wind Tunnel located in a cold climate chamber, we assess the ability of the fabricated coatings to reduce the ice accretion on aluminum substrates in both glaze and rime icing regimes. The reduction of ice adhesion was instead determined with the Double Lap Shear Test. The dynamic behavior of droplets was also evaluated via goniometric contact angle hysteresis calculation, at both room and sub-zero temperatures (Figure 1).

The remarkable icephobic behavior of SLIPS coatings developed highlights their potential as candidate for passive IPS especially in those application fields in which dynamic water repellency at subzero temperature is crucial (e.g. aeronautics). Moreover, in order to achieve a durable water repellency even when exposed at sub-zero temperature, SLIPS design proved the ability to recover functional performance after oil depletion phenomena.

**Keywords:** coatings/surfaces with reduced ice adhesion, anti-icing coatings and surfaces, bio-

inspired coatings for anti-icing applications, nanostructured surfaces against icing



**Figure 1:** below-zero Contact Angle Hysteresis (CAH) grouped by coating type

## References:

1. Meuler, A.J., Smith, J.D., Varanasi, K.K., Mabry, J.M., McKinley, G.H., Cohen, R.E. (2012) Relationships between Water Wettability and Ice Adhesion, *ACS Applied Materials and Interfaces*, 2, 3100.
2. Subramanyam, S.B., Rykaczewski, K., Varanasi, K.K. (2013) Ice Adhesion on Lubricant-Impregnated Textured Surfaces *Langmuir*, 29, 13414.
3. Boveri, G., Corozzi, A., Veronesi, F., Raimondo, M. (2021) Different Approaches to Low-Wettable Materials for Freezing Environments: Design, Performance and Durability, *Coatings*, 11, 77.

# Combination of Coagulation, Adsorption, and Ultrafiltration Processes for Organic Matter Removal from Peat Water

M Elma <sup>1,2,\*</sup>, A E Pratiwi <sup>1,2</sup>, A Rahma <sup>2</sup>, E L A Rampun <sup>2</sup>, Mahmud <sup>3,\*</sup>, C Abdi <sup>3</sup>, R Rosadi <sup>2,4</sup>, D H Y Yanto <sup>5</sup> and M R Bilad <sup>6</sup>

<sup>1</sup>Department of Chemical Engineering, Faculty of Engineering, Lambung Mangkurat University, Jl. A. Yani KM 36 Banjarbaru 70714, Indonesia

<sup>2</sup>Materials and Membranes Research Group (M2ReG), Lambung Mangkurat University, Jl. A. Yani KM 36 Banjarbaru 70714, Indonesia

<sup>3</sup>Department of Environment Engineering, Faculty of Engineering, Lambung Mangkurat University, Jl. A. Yani KM 36 Banjarbaru 70714, Indonesia

<sup>4</sup>Postgraduate Program, Department of Natural Resource and Environmental Management, Lambung Mangkurat University, Jl. A. Yani KM 36 Banjarbaru 70714, Indonesia

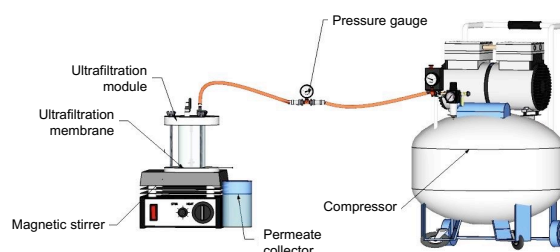
<sup>5</sup>Research Center for Biomaterials, National Research and Innovation Agency (BRIN), Jl. Raya Bogor KM 46, Cibinong Science Center, Cibinong, Bogor 16911, Indonesia

<sup>6</sup>Faculty of Integrated Technologies, Universiti Brunei Darussalam, Gadong BE1410, Brunei

## Abstract:

The high content of natural organic matter (NOM) is one of the challenging characteristics of peat water. It is also highly contaminated and contributes to some water-borne diseases. Before being used for potable purposes, peat water must undergo a series of treatments, particularly for NOM removal. This study investigated the effect of coagulation using aluminum sulfate coagulant and adsorption using powdered activated carbon (PAC) as a pretreatment of ultrafiltration (UF) for removal of NOM from actual peat water. After preparation and characterization of polysulfone (Psf)-based membrane, the system's performance was evaluated using actual peat water, particularly on NOM removal and the UF performances. The coagulation and adsorption tests were done under variable dosings. Results show that pretreatment through coagulation-adsorption successfully removed most of the NOM. As such, the UF fouling propensity of the pretreated peat water was substantially lowered. The optimum aluminum sulfate dosing of 175 mg/L as the first pretreatment stage removed up to 75–78% NOM. Further treatment using the PAC-based adsorption process further increased 92–96% NOM removals at an optimum PAC dosing of 120 mg/L. The final UF-PSf treatment reached NOM removals of 95% with high filtration fluxes of up to 92.4 L/(m<sup>2</sup>.h). The combination of three treatment stages showed enhanced UF performance thanks to partial pre-removal of NOM that otherwise might cause severe membrane fouling.

**Keywords:** coagulation-adsorption; membrane; organic matter; peat water; ultrafiltration; polysulfone.



**Figure 1:** Illustration of the ultrafiltration dead-end experimental setup.

## References:

1. Tang, C.; He, Z.; Zhao, F.; Liang, X.; Li, Z. Effects of Cations on The Formation of Ultrafiltration Membrane Fouling Layers When filtering Fulvic Acid. *Desalination* **2014**, *352*, 174–180. <https://doi.org/10.1016/j.desal.2014.08.020>.
2. Matilainen, A.; Vepsäläinen, M.; Sillanpää, M. Natural organic matter removal by coagulation during drinking water treatment: A review. *Adv. Colloid Interface Sci.* **2010**, *159*, 189–197.
3. Menya, E.; Olupot, P.; Storz, H.; Lubwama, M.; Kiros, Y. Production and performance of activated carbon from rice husks for removal of natural organic matter from water: A review. *Chem. Eng. Res. Des.* **2018**, *129*, 271–296.
4. McMeen, C.R.; Benjamin, M.M. NOM removal by slow sand filtration through iron oxide-coated olivine. *J. Am. Water Work. Assoc.* **1997**, *89*, 57–71.

# Control of metal ion release from magnetron sputtered Ag-Cu coatings on textiles

Serdar Sonay Özbay<sup>1,2,\*</sup>, Golnaz Taghavi Pourian Azar<sup>1</sup>, Julie Sharp<sup>2</sup>, Gayathri Devi Rajmohan<sup>2</sup>, Andrew Cobley<sup>1</sup>

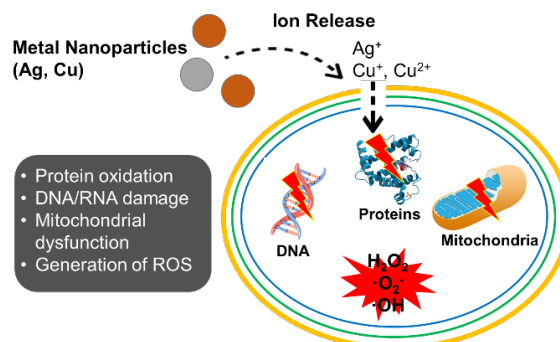
<sup>1</sup> Faculty of Engineering Computing and Environment, Coventry University, Coventry, UK

<sup>2</sup> Institute for Frontier Materials, Deakin University, VIC, Australia

## Abstract:

Ag and Cu nanoparticles (NPs) are widely used as antibacterial agents to functionalise textile surfaces to fight antibiotic-resistant bacteria and healthcare-acquired infections (HAI) in hospitals. Although the precise antibacterial mechanisms of NPs are still being investigated, they mainly deactivate bacteria by releasing metal ions that penetrate bacterial cells and disrupt the intercellular content (Figure 1). It is known that different bacteria species and strains show different susceptibilities to Ag and Cu NPs. However, it is not possible to draw definitive comparisons between the antibacterial efficacy of Ag and Cu NPs due to the conflicting studies. Nonetheless, the latest studies have shown consistent results on Ag-Cu NP mixtures showing superior antibacterial properties compared to their pure alternatives. Combining Ag-Cu NPs and finely tuning the release of Ag and Cu ions will let us formulate a coating for a specific bacterial species or environment. Our study focuses on controlling the metal ion release from Ag-Cu NPs deposited on PET textiles by the magnetron sputtering technique. Our preliminary studies have shown that the ratio of released Cu/Ag ion concentration reaches 50 when the Ag<sub>50</sub>-Cu<sub>50</sub> NP mixture is used. In this study, we report the effect of non-toxic alloying elements, such as magnesium, and the elemental composition on the metal ion release behaviour of Ag, Cu and Ag-Cu NPs. In addition to the metal ion release, electrochemical, optical and electrical properties of multi-element (AgCu, AgMg, CuMg, AgCuMg) NP mixture systems have been investigated.

**Keywords:** antibacterial, nanoparticles, silver, copper, magnesium, ion release, galvanic corrosion, hospital-acquired infections.



**Figure 1:** A general antibacterial mechanism for metal ion release of Ag and Cu NPs.

## References:

1. Eremenko, A. M., Petrik, I. S., Smirnova, N. P., Rudenko, A. V., & Marikvas, Y. S. (2016). Antibacterial and Antimycotic Activity of Cotton Fabrics, Impregnated with Silver and Binary Silver/Copper Nanoparticles. *Nanoscale Res Lett*, 11(1), 28.
2. Rtimi, S., Sanjines, R., Pulgarin, C., & Kiwi, J. (2016). Microstructure of Cu–Ag Uniform Nanoparticulate Films on Polyurethane 3D Catheters: Surface Properties. *ACS Applied Materials & Interfaces*, 8(1), 56-63.



# Atmospheric pressure plasma superhydrophobic bilayer coatings to limit dairy fouling adhesion

M. Saget<sup>1,2</sup>, N. Nuns<sup>1</sup>, L. Azevedo-Scudeller<sup>1</sup>, G. Delaplace<sup>1</sup>, V. Thomy<sup>2</sup>, Y. Coffinier<sup>2</sup>, M. Jimenez<sup>1,3\*</sup>

<sup>1</sup>Univ. Lille, CNRS, INRA, ENSCL, UMR 8207 - UMET - Unité Matériaux et Transformations, F-59000 Lille, France

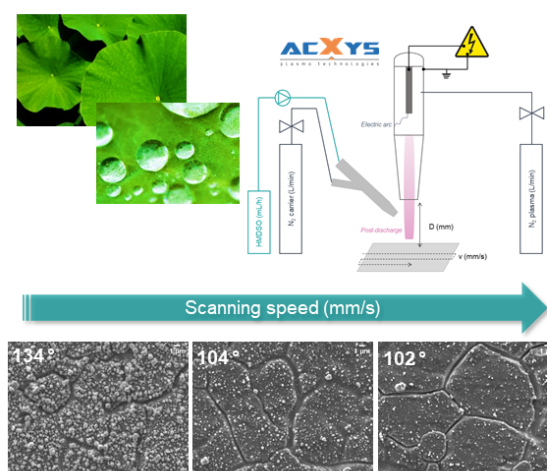
<sup>2</sup>Université de Lille, Institute of Electronics, Microelectronics and Nanotechnology (IEMN), UMR 8520, F-59000 Lille, France

<sup>3</sup>Institut Universitaire de France, Paris, France

## Abstract:

Unwanted fouling deposits are formed on heat exchangers' surface during mandatory thermal treatments (pasteurization, sterilization) of dairy products in food processing industries. These deposits can contaminate dairy products to pasteurize and also impair heat transfer mechanisms by creating a thermal resistance, thus leading to regular shut down of the processes. Therefore, periodic and drastic cleaning-in-place (CIP) procedures are implemented. These CIP involve the use of chemicals and high amount of water, thus increasing environmental burden. It has been estimated that 80% of production costs are owed to dairy fouling deposit. To reduce dairy fouling, two pathways have been considered: (i) Process conditions optimization, mainly tested by food-processing industries and (ii) Stainless steel surface anti-fouling coating to either inhibit attachment of depositing species or to ease their removal during cleaning. Here, we focus on this latter approach, by developing specific coatings (slippery liquid-infused porous surfaces (SLIPS) and atmospheric plasma coatings) of low contact angle hysteresis to limit fouling adhesion onto stainless steel surfaces. Superhydrophobic and stable bilayers based on fluoro and silane coatings were deposited by atmospheric pressure plasma (Lab-Scan, Axcys Technologies). ToF-SIMS depth profile analyses allowed to confirm the deposition of two distinct layers. Encouraging fouling-release (*i.e.* ease of fouling removal) performances were obtained (fouling deposit reduction of 72%).

**Keywords:** Hydrophobic nano-structured coating, Anti-fouling, Biomimetic, Dairy processing



**Figure 1:** Figure illustrating the atmospheric pressure plasma process used to deposit hydrophobic and superhydrophobic surfaces. SEM images show resulting hydrophobic nano-structured plasma.

## References:

1. van Asselt, A. J.; Vissers, M. M.; Smit, F.; De Jong, P. (2005) In-line control of fouling. Proceedings of heat exchanger fouling and cleaning-challenges and opportunities. Engineering Conferences International Kloster Irsee, Germany
2. Zouaghi, S.; Six, T.; Bellayer, S.; Moradi, S.; Hatzikiriakos, S. G.; Dargent, T.; Thomy, V.; Coffinier, Y.; André, C.; Delaplace, G.; Jimenez, M. (2017) Antifouling Biomimetic Liquid-Infused Stainless Steel: Application to Dairy Industrial Processing. *ACS Appl. Mater. Interfaces*, 9, 26565–26573
3. Zouaghi, S.; Six, T.; Bellayer, S.; Coffinier, Y.; Abdallah, M.; Chihib, N-E.; André, C.; Delaplace, G.; Jimenez, M. (2018) Atmospheric pressure plasma spraying of silane-based coatings targeting whey protein fouling and bacterial adhesion management. *Applied Surface Science*, 455, 392–402

## Evaluation of galvanic anodes capacity as per NACE TM0190-98 test methods

Nayif ALRasheedi<sup>1</sup>, Sultan ALMutairi<sup>2</sup>

<sup>1</sup> Saudi Aramco, Saudi Arabia

<sup>2</sup> Aramco, Saudi Arabia

### **Abstract:**

NACE-TM-0190 method was implemented to evaluate Aluminum, Zinc & High Temperature Zinc galvanic anodes used for cathodic protection system. The test was conducted by passing a fixed direct current through test cells electrically connected in series. The test cell is made of carbon steel and it is the cathode in the electrical circuit. Each test cell contains an electrolyte for current flow which is seawater. The fixed direct current flows for a period of 14 days during which the anodes are partially consumed. The ampere-hour obtained per unit mass of anode lost is calculated. The value is compared to that of pure anode metal to determine the percentage efficiency of each tested galvanic anode.

This paper presents new chemical composition of galvanic anodes that have not been used before

# Breakdown criteria in air : critical analysis and applications

O. Eichwald <sup>1</sup>, O. Ducasse <sup>1</sup> and M. Yousfi <sup>1</sup>

<sup>1</sup> LAPLACE, Université de Toulouse, CNRS, INPT, UPS, France

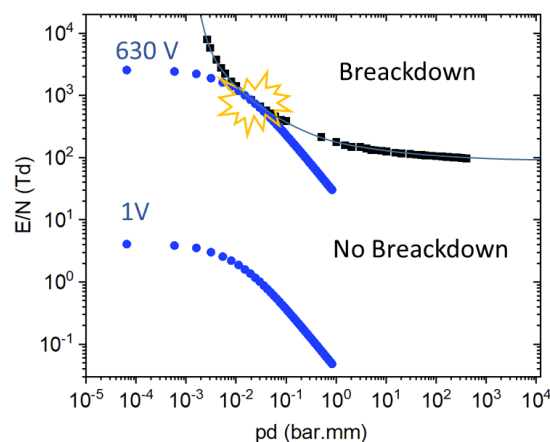
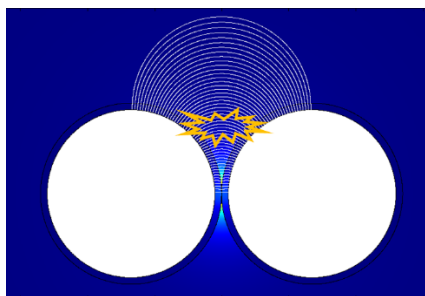
## Abstract:

With the development of more electric ground and air transportation, the replacement of greenhouse gases in circuit breakers, the increase of voltage in power electronics or the understanding of how to initiate and maintain an electrical discharge in a plasma device, the study of the breakdown criteria<sup>1,2,3</sup> becomes essential to anticipate risks of material degradations which can occur additionally at different pressure levels. Breakdown voltages are often extracted from experimental Paschen curves (that give the breakdown voltage as a function of the product pressure distance). Several criteria have also been proposed for different pressure ranges (Townsend's criterion, Meek and Craggs' criterion, Pedersen's criterion, and so one).

Even though these curves and criteria exist for a long time, many questions remain: How can the Paschen curves and the various criteria be carefully interpreted and used? What is the difference between a critical electric field and a breakdown electric field? Should we work with the potential, electric field, reduced electric field, gas pressure or its density?

It is proposed to answer these questions by analysing the known data in air, relying on both experimental and simulation results and give concrete samples of how to exploit a redifined Paschen curve to determinate the breakdown voltage between 2 insulated wires (Figure 1) or between 2 metal spheres for different gas pressures in air.

**Keywords:** Plasma, Discharge, Breakdown criteria, Paschen curve, Critical electric field.



**Figure 1:** Mean reduced electric field along each field strength lines (blue points) for several applied voltage between 2 insulated wires compared with the reduced breakdown electric field.

## References:

1. O. Eichwald, M. Yousfi and O. Ducasse, Breakdown criteria in air : an overview supported by simulation predictions, 11<sup>ème</sup> conférence de la Société Française d'Electrostatique, 29-31 août 2018, Grenoble, France
2. TW. Dakin, Breakdown of gases in uniform field Paschen curves for Nitrogen, Air and SF<sub>6</sub>, *Electra*, N° 62, 1974
3. Pedersen, On the Electrical Breakdown of Gaseous Dielectrics, *IEEE Transactions on Electrical Insulation*, vol. 24, n°5, 1989
4. Eichwald, H Bensaad, O Ducasse and M Yousfi, Effects of numerical and physical anisotropic diffusion on branching phenomena of negative-streamer dynamics, *J. Phys. D: Appl. Phys.* 45 (2012) 385203 (15pp)

# Active Janus Particles in a Complex Plasma

V. Nosenko<sup>1</sup>

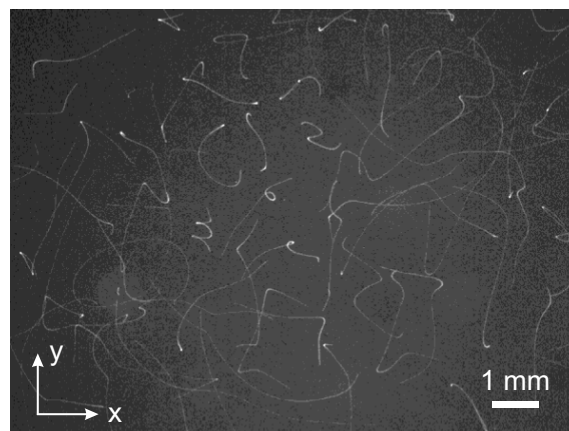
<sup>1</sup> Institut für Materialphysik im Weltraum, Deutsches Zentrum für Luft- und Raumfahrt (DLR), D-51147 Cologne, Germany

## Abstract:

A complex plasma is a suspension of micron-size solid particles in regular plasma. Complex plasmas are excellent model systems which are used to study various plasma-specific and generic phenomena at the level of individual particles. Their advantages include the possibility of direct and real-time observation of virtually undamped dynamics of the particle suspended in a rarefied gas. Active matter is a collection of active particles, which can convert the energy coming from their environment into directed motion, therefore driving the whole system far from equilibrium. Active matter has some intriguing physical properties and potentially a number of applications in nanotechnologies, biology, and medicine. It has recently become a hot topic of multiple interdisciplinary studies.

A prominent example of particles that can be active in various environments is the so-called Janus particles, which have two sides with different properties. Janus particles where two sides were made of (or coated with) different metals were previously found to be active in certain aqueous solutions. In this presentation, we report on our experiments with Janus particles - polymer microspheres where one half was plasma-coated with a thin layer of platinum - suspended in a gas discharge plasma [1]. Our experimental setup was a modified Gaseous Electronics Conference (GEC) radio-frequency (rf) reference cell. Plasma was produced by an rf capacitively coupled discharge at 13.56 MHz in argon. The gas pressure was varied in the range of 0.66-13.3 Pa, the rf discharge power was in the range of 1-20 W. When suspended in plasma, the Janus particles moved in characteristic looped trajectories suggesting a combination of spinning and circling motion; their interactions led to the emergence of rich dynamics characterized by non-Maxwellian velocity distribution. The particle propulsion mechanism is discussed, the force driving the particle motion is identified as a combination of photophoretic force and asymmetric ion-drag force due to the plasma-particle surface interaction.

**Keywords:** complex plasma, active matter, Janus particles, plasma coating.



**Figure 1:** Trajectories of Janus particles suspended as a 2D layer in rf plasma sheath. 545 frames of the top-view video (during 2.18 s) were superposed, the brightness and contrast were adjusted for better viewing. Note a characteristic curly appearance of many trajectories. The argon pressure was 0.66 Pa, the rf discharge power was 20 W.

## References:

1. Nosenko, V., Luoni, F., Kaouk, A., Rubin-Zuzic, M., and Thomas, H. (2020), Active Janus particles in a complex plasma, *Phys. Rev. Research*, 2, 033226.

# Plasma based CO<sub>2</sub> Utilization – A Comparison

S. Renninger <sup>1\*</sup>, P. Rößner <sup>1</sup>, J. Stein <sup>1</sup>, M. Lambarth <sup>1</sup>, P. Birke <sup>1</sup>

<sup>1</sup> Electrical Energy Storage Systems; Institute for Photovoltaics; University of Stuttgart, Pfaffenwaldring 47, 70569 Stuttgart, Germany

\*Correspondence: stephan.renninger@ipv.uni-stuttgart.com; Tel.: +4971168561508

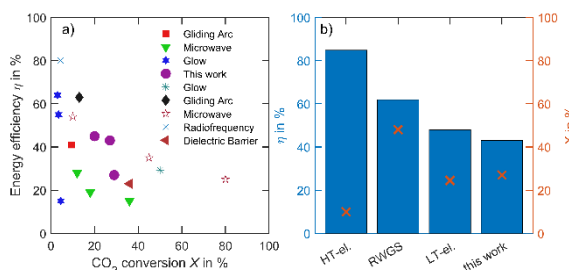
## Abstract:

Atmospheric CO<sub>2</sub> levels pose one of the biggest challenges for our future. CO<sub>2</sub> utilization to value added compounds is an attractive solution. Different fields offer solutions for energy efficient CO<sub>2</sub> utilization. Among the most promising are electrochemistry, thermochemistry and plasma catalysis. In this (poster/presentation), different approaches are compared: High- and low temperature electrolysis<sup>1,2</sup>, reverse water gas shift<sup>3</sup> and plasma based processes<sup>4</sup> can all reduce CO<sub>2</sub> into carbon monoxide.

We researched cutting edge literature for the most promising contenders in each category. Energy efficiency and CO<sub>2</sub> conversion are calculated and compared. The main focus are various plasma technologies at ambient pressure. Microwave, radio frequency, dielectric barrier discharge, gliding arc and glow discharge reactors are presented. One among them is our recently published glow discharge reactor, that utilizes a magnetic field to improve the sweeping of the working gas<sup>5</sup>. Figure 1 (a) shows the energy efficiency over the CO<sub>2</sub> for the different plasma approaches published in literature. Figure 1 (b) compares the atmospheric pressure glow discharge reactor from our group to other approaches not based on plasma.

In general, the energy efficiency of plasma-based systems is gaining ground compared to competing technologies such as electrolysis and thermochemical approaches. Focus thus shifts to scalability, lifespan and, most importantly, integration. After all, none of the presented technologies manage to produce pure product gases; their separation is a major task for which few technologies are available. The integration of electrochemical oxygen pumps or separation membranes into plasma reactor systems will be a future focus. Generally, we argue in favour of a broad portfolio of technological solutions for the future of CO<sub>2</sub> utilization. The use cases are as diverse as the solutions and detailed analysis is necessary to reveal the ideal solution depending on the framework conditions. Our results illustrate that plasma technology can play an important role in CO<sub>2</sub> utilization, which is a cornerstone of a fossil-free economy.

**Keywords:** plasma catalysis; CO<sub>2</sub> splitting; glow discharge; energy efficiency; CO<sub>2</sub> electrolysis; CO<sub>2</sub> utilization; magnetic field; power-to-X



**Figure 1:** A comparison of the achieved energy efficiency  $\eta$  and conversion  $X$  for CO<sub>2</sub> splitting by plasma is shown in (a) (References not included in this abstract, see <sup>5</sup> for details). In (b) the results are compared to high temperature electrolysis (HT-el.<sup>1</sup>), low temperature electrolysis (LT-el.<sup>2</sup>) and reverse water gas shift (RWGS<sup>3</sup>).

## References:

1. Kaur G, Kulkarni AP, Giddey S. CO<sub>2</sub> reduction in a solid oxide electrolysis cell with a ceramic composite cathode: Effect of load and thermal cycling. *Int J Hydrogen Energy*. Published online 2018;21769-21776. doi:10.1016/j.ijhydene.2018.10.014
2. Liu Z, Yang H, Kutz R, Masel RI. CO<sub>2</sub> Electrolysis to CO and O<sub>2</sub> at High Selectivity, Stability and Efficiency Using Sustainion Membranes. *J Electrochem Soc*. 2018;165(15):J3371-J3377. doi:10.1149/2.0501815jes
3. Zonetti PC, Letichevsky S, Gaspar AB, Sousa-Aguiar EF, Appel LG. The Ni<sub>0.75</sub>Ce<sub>0.25</sub>Zr<sub>0.25</sub>-xO<sub>2</sub> solid solution and the RWGS. *Appl Catal A Gen*. 2014;475:48-54. doi:10.1016/j.apcata.2014.01.004
4. Renninger S, Lambarth M, Birke KP. High efficiency CO<sub>2</sub>-splitting in atmospheric pressure glow discharge. *J CO<sub>2</sub> Util*. 2020;42(September):101322. doi:10.1016/j.jcou.2020.101322
5. Renninger S, Rößner P, Stein J, Lambarth M, Birke KP. Towards High Efficiency CO<sub>2</sub> Utilization by Glow Discharge Plasma. *Processes*. 2021;9(11):2063. doi:10.3390/pr9112063

# Investigation of Temporal Evolution of Vacuum Arc with Heated Cathode Made of Cerium Dioxide

Melnikov A.D., Usmanov R.A., Polishchuk V.P., Antonov N.N., Gavrikov A.V., Liziakin G.D.  
Joint Institute for High Temperatures of the Russian Academy of Sciences, Moscow, Russia

## Abstract:

To date the development of plasma sources generating dielectric materials plasma flow is relevant for the wide-spread technologies of functional coatings formation [1] and for specific research fields such as plasma separation methods [2]. Utilizing of plasma sources based on vacuum arcs allows to provide high performance, meanwhile the parameters of the generated plasma flow depend on the type of cathode attachment being realized under particular experimental conditions [3]. We investigated a vacuum arc with a heated cathode and its material was cerium dioxide due to its application for creation corrosion-resistant coatings and ultraviolet emission protection films [4-5]. A vacuum arc on a cathode made of cerium dioxide was described in [2]. This work is devoted to the study of the temporal evolution of discharge and the dependence of plasma composition on parameters of arc.

The discharge was initiated in a vacuum chamber with a volume of  $\sim 1 \text{ m}^3$ , the pressure of residual gases was about  $10^{-5} \text{ Pa}$  [2]. The  $\text{CeO}_2$  cathode weighing about 5 g was placed in a molybdenum and tungsten crucible. The crucible heating was carried out by means of 1.5 kW electron beam heating. The temperature of the crucible side surface was measured using an optical pyrometer. To establish the ionic composition, a time-of-flight mass spectrometer with a resolution of 25 located behind the anode at a distance of 300 mm was applied. The voltage, mass-spectrum of plasma ions, cathode temperature and emission intensity were continuously recorded during the experiments. The discharge current was varied from 40 to 90 A, and the voltage and crucible temperature ranging from 10 to 14 V and from 2.15 to 2.35 kK respectively.

Fluctuations of arc voltage, amplitudes of mass peaks and spectral lines intensity were detected. The relative values of fluctuations vary over time in the range from 2 % to 10 % for voltage and from 10 % to 30 % for the amplitudes of mass peaks. The average value of the voltage at fixed arc current and the crucible temperature was decreasing during the experiment by up to 1.5 V. Unstable regimes were observed, remaining from several seconds to several minutes. The amplitude of fluctuations was increasing, ions of

higher multiplicities were observing and oscillations of the plasma column were recording within such regimes. The video recording of the cathode surface revealed that during the experiments both vacuum arc modes without cathode spots and with type 2 spots occurred [6]. The transitions between modes also occurred in case of the varying of crucible temperature. After a time of about 1000 s after initiation, the discharge became less stable. The study of the ionic composition revealed that single cerium ions  $\text{Ce}^+$  and molecular ions  $\text{CeO}^+$  predominate in the plasma flow, and molybdenum (tungsten) ions  $\text{Mo}^+$  ( $\text{W}^+$ ) are also present. The ionic composition of the plasma changes over experiment time, even within stable discharge regime: the content of crucible material ions in the plasma flow changes, while the intensities of the  $\text{Ce}^+$  and  $\text{CeO}^+$  peaks are approximately constant. Furthermore the densities of plasma flow ionised components at different arc current values was measured via precalibrated TOF mass-spectrometer. Therefore the influence of arc current value was investigated. Finally, all the experiments was carried out for two crucible materials (molybdenum and tungsten) with the aim to reveal its possible affection on discharge parameters. In the result less stability and higher crucible material content in plasma were observed in the case of tungsten crucible.

The investigation was supported by the Russian Science Foundation, Grant No. 21-72-00077, <https://rscf.ru/en/project/21-72-00077/>

**Keywords:** plasma sources, vacuum arc, temporal evolution, plasma composition

## References:

1. Liang H., Ming F., Alshareef H., *Advanced Energy Materials*, 2018, V. 8, P. 1801804.
2. Usmanov R.A. et. al., *Physics of Plasmas*, 2018, V. 25, P. 063524.
3. V. P. Polishchuk et. al., *High Temperature*, 2020, V. 58, N. 4, P. 476–494.
4. Ershov S., et. al., *Corrosion Science* 2013, V. 75, P. 158–168.
5. Balakrishnan G., Sudhakara P., Abdul Wasy, Ha Sun Ho, Shin K.S., Song J.I., *Thin Solid Films* 2013, V. 546, P. 467–471.
6. Juttner B., *J. Phys. D: Appl. Phys.*, 2001, V. 34, R103–R123.



## Pb+Ag mixture space separation in crossed fields of background discharge at the LaPlaS setup

G. Liziakin<sup>1\*</sup>, N. Antonov<sup>1</sup>, A. Gavrikov<sup>1</sup>, A. Oiler<sup>1,2</sup>, A. Melnikov<sup>1,2</sup>, V. Smirnov<sup>1</sup>, R. Timirkhanov<sup>1</sup>, R. Usmanov<sup>1</sup>, L. Volkov<sup>1,2</sup>, N. Vorona<sup>1</sup>,

<sup>1</sup> Joint Institute for High Temperatures of the Russian Academy of Sciences, Moscow, Russia

<sup>2</sup> Moscow Institute of Physics and Technology (National Research University), Moscow, Russia

### Abstract:

The plasma separation technique is one of the dry methods for spent nuclear fuel (SNF) reprocessing. The main task of this process is to separate actinides from fission products. Over the past two decades this area has seen intensive development, which resulted in number of various separating schemes were proposed [1,2]. All of them imply the stage of transfer solid state SNF into the plasma state. Influenced by electric and magnetic fields the space separation by mass occurs. The fundamental difference between plasma-based devices and electromagnetic separators is that the ions of the separated elements move in the conditions of the compensated space charge. Thus restriction on the ionic current value (Child–Langmuir law) are removed and the productivity rate required for its practical application in the nuclear industry can be achieved.

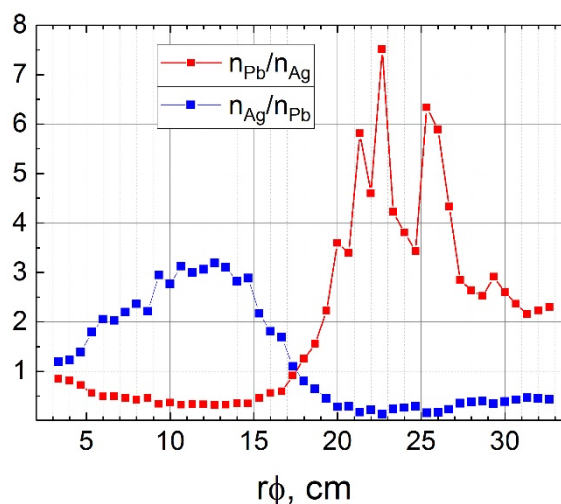
In the LaPlaS installation 4 coils solenoid creates an axial magnetic field in a cylindrical vacuum chamber with the diameter of 85.6 cm and the length of 220 cm. A radial electric field is created by a reflex discharge with thermionic cathode. The solid Pb+Ag mixture is transferred into the plasma state by an arc plasma source [3]. Thus, in the chamber 2 discharges operate simultaneously; background reflex discharge which creates the radial electric field and the arc discharge which serves as a source of separated plasma. A metal substrate is coated with ionized beams of Pb and Ag. The samples of substrate are analyzed by energy-dispersive X-ray spectroscopy.

In this presentation, we provide results of Pb+Ag mixture separation.

### Acknowledgments:

This study was supported by the Russian Science Foundation № 21-19-00716,  
<https://rscf.ru/en/project/21-19-00716/>.

**Keywords:** plasma mass separation, reflex discharge, ExB plasmas.



**Figure 1:** Distribution of lead and silver atoms on the substrate.

### References:

1. Dolgolenko D A and Muromkin Y A 2017 Separation of mixtures of chemical elements in plasma Phys.-Usp. 60 994.
2. Zweben S J, Gueroult R and Fisch N J 2018 Plasma mass separation Phys. Plasmas 25 90901.
3. Antonov N, Liziakin G, Usmanov R, Gavrikov A, Vorona N and Smirnov V 2018 The influence of reflex discharge electric field on propagation of injected lead plasma jet Phys. Plasmas 25 123506.
4. Liziakin G, Antonov N, Smirnov V S et.al., 2021 Plasma mass separation in configuration with potential well, J Phys. D Appl. Phys., 54, 414005.
5. Liziakin G, Antonov N, Usmanov R et.al. 2021 Experimental demonstration of plasma mass separation in a configuration with a potential well and crossed electric and magnetic fields Plasma Phys. Control. Fusion 63 032002.

# Optimization of PECVD SiO<sub>x</sub> passivation of GaN for interface charge control

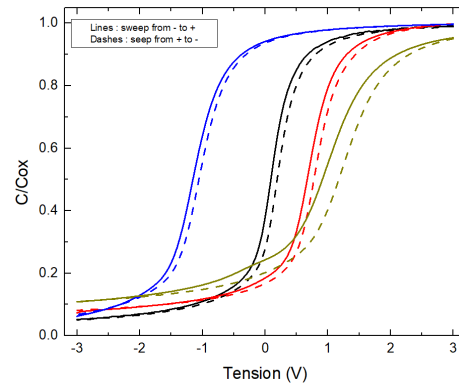
O. Richard, V. Aimez, A. Jaouad

Institut Interdisciplinaire d'Innovation Technologique (3IT), Université de Sherbrooke, Sherbrooke, Québec, Canada

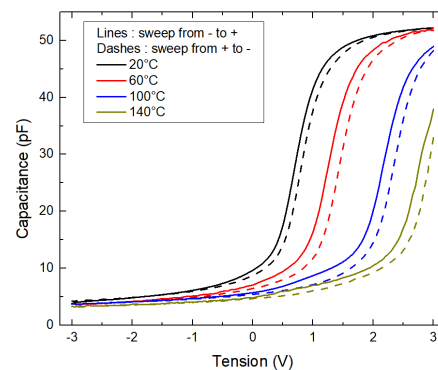
## Abstract:

Heterostructure field effect transistors (HFET) based on GaN have a great potential for high frequency and high power applications. By virtue of having an inherent bidimensionnal electron gas as their channel at the GaN/AlGaN interface, these devices are normally-on (conducting channel under no gate bias). We presented in the past our results on the study of PECVD parameters for (0001) n-GaN passivation by SiO<sub>x</sub> deposition. We had shown in particular that low power operation with an increased SiH<sub>4</sub>/N<sub>2</sub>O ratio could be used to obtain a negative charge density at the SiO<sub>x</sub>/GaN interface. This deposition process could therefore be of interest for the design of normally-off (positive threshold voltage) HFETs. In the present study, we first confirm the trends previously found. MIS capacitors were fabricated in a study of the following PECVD parameters : SiH<sub>4</sub>/N<sub>2</sub>O ratio, RF power, pressure and temperature. C-V measurements (fig. 1) again demonstrate that these parameters can be used to alter the flatband voltage considerably. Of most interest is the red curve obtained with a low power and high SiH<sub>4</sub>/N<sub>2</sub>O ratio deposition process. The curve is shifted towards positive voltage values, while presenting low hysteresis and density of surface states. Furthermore, initial characterization at higher temperatures (fig. 2) shows a much more stable operation than before. No marked deformations of C-V curves appear this time and the increase of hysteresis, although present, seems to saturate over 60°C. FTIR chemical analysis of similarly deposited layers is under way as well as advanced interface characterization by C-V measurements under UV light.

**Keywords:** GaN, PECVD, passivation, MIS devices, normally-off, HFET



**Figure 1 :** Example of 1MHz C-V measurements of Al/SiO<sub>x</sub>/GaN MIS capacitors with 20-30 nm SiO<sub>x</sub>. Only the parameters of PECVD vary. Note the wide flatband voltage variations.



**Figure 2 :** 1 MHz C-V measurements of Al/SiO<sub>x</sub>/GaN MIS capacitor at elevated temperatures. The device from which results are obtained corresponds to the red curve in fig. 1. The positive shift with increasing temperature is due to the pyroelectric effect.

## References:

1. Chakroun, A., Jaouad, A., Soltani, A., Arenas, O., Aimez, V., Arès, R., Maher, H. (2017), "AlGaN/GaN MOS-HEMT Device Fabricated Using a High Quality PECVD Passivation Process", IEEE Electron Device Lett. Vol. 38 No. 6, 779-782
2. Lu, X., Yu, K., Huaxing, J., Zhang, A., Lau, K.M. (2017), "Study of Interface Traps in Al-GaN/GaN MISHEMTs Using LPCVD SiNx as Gate Dielectric", IEEE Trans. Electron Devices Vol. 64 No. 3, 824-830
3. Sayed, I., Liu, W., Chan, S., Gupta, C., Guidry, M., Li, H., Keller, S., Mishra, U. (2019), "Net Negative Interface Charge for Si<sub>3</sub>N<sub>4</sub> and SiO<sub>2</sub> Grown on 000-1 Polar GaN", App. Phys. Lett. 115, 032103

# Dynamic properties of dust particles in a DC glow discharge in an external magnetic field

A.R. Abdirakhmanov<sup>1,2,\*</sup>, N.Kh. Bastykova<sup>1</sup>, S.Kh. Kodanova<sup>1</sup>, T.S. Ramazanov<sup>1</sup>

<sup>1</sup>IETP, Al-Farabi Kazakh National University, Almaty, Kazakhstan

<sup>2</sup>Laboratory Engineering Profile, Al-Farabi Kazakh National University, Almaty, Kazakhstan

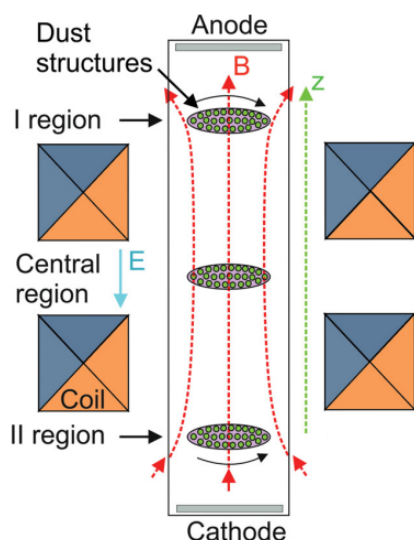
## Abstract:

The study of the effect of a magnetic field on a plasma system is of great interest, which plays a crucial role in thermonuclear and astrophysical plasmas. The study of the effect of a magnetic field on a plasma system is of great interest, which plays a crucial role in thermonuclear and astrophysical plasmas.

In the presented work, a complex plasma in a DC glow discharge at low pressures was investigated. The source of the magnetic field was a Helmholtz coil system where a uniform field is generated in the middle and a nonuniform field in the upper and lower regions. It was considered in different magnetic field regions the dynamics of micron-sized particles that is located in the lateral branches of the glass discharge tube where a glow discharge is generated. The dynamics of micron particles were captured on a video camera and their behavior was analyzed using the particle image velocimetry (PIV) method at different magnetic field inductions. A theoretical model

was proposed to explain the experimental observations.

**Keywords:** DC glow discharge, magnetic field, complex (dusty) plasma, dust structures



**Figure 1:** . Illustration of the experimental setup

## References:

1. Abdirakhmanov, A. R., Moldabekov, Z. A., Kodanova, S. K., Dosbolayev, M. K., & Ramazanov, T. S. (2019) . Rotation of dust structures in a magnetic field in a dc glow discharge, *IEEE Transactions on Plasma Science*, 47, 3036–3040
2. Abdirakhmanov, A. R., Bastykova, N. K., Kodanova, S. K., & Ramazanov, T. S. (2021), Rotation of dust particles in an inhomogeneous weak magnetic field in a dc glow discharge, *Physics of Plasmas*, 28(7), 074503.
3. Kodanova, S., Abdirakhmanov, A., D'yachkov, L., Bastykova, N., & Ramazanov, T.S. (2022), Ring dust structures in a weak inhomogeneous magnetic field, *Contributions to Plasma Physics*, e202100254.
4. Abdirakhmanov, A. R., Karasev, V. Y., Dzlieva, E. S., Pavlov, S. I., Novikov, L. A., Dosbolayev, M., Kodanova, S., Ramazanov, T. S (2021) Rotation of the dust structure in a strong non-uniform magnetic field, *High Temperature*, 59(5), 657–662.

## Plasma assisted CO<sub>2</sub> dissociation in pure and gas mixture streams at ambient conditions

P. Navascués<sup>1</sup>, M Oliva-Ramírez<sup>2</sup>, J. Cotrino<sup>1,2</sup>, A. R. González-Elípe<sup>1</sup>, A. Gómez-Ramírez<sup>1,2\*</sup>

<sup>1</sup>Laboratory of Nanotechnology on Surfaces and Plasma, Instituto de Ciencia de Materiales de Sevilla (CSIC-Universidad de Sevilla), C/ Américo Vespucio 49, Sevilla, 41092, Spain

<sup>2</sup>Departamento de Física Atómica, Molecular y Nuclear, Universidad de Sevilla, Avda. Reina Mercedes, Sevilla 41012, Spain

### Abstract:

Carbon dioxide decomposition is a challenging target to combat climate change. Nonthermal plasmas are advantageous for this purpose because they operate at ambient conditions and can be easily scaled-up. In this study, we attempt the CO<sub>2</sub> splitting into CO and O<sub>2</sub> in a parallel plate packed-bed plasma reactor moderated with Lead Zirconate Titanate (PZT) as ferroelectric component, achieving conversion rates and energy efficiencies higher than those obtained with BaTiO<sub>3</sub> in our experimental device. The analysis of the reaction mechanisms with optical emission spectroscopy under various operating conditions has shown a direct correlation between energy efficiency and intensity of CO\* emission bands. These results and those obtained with a LiNbO<sub>3</sub> plate placed onto the active electrode suggest that high temperature electrons contribute to the splitting of CO<sub>2</sub> through an enhancement in the formation of CO<sub>2</sub><sup>+</sup> intermediate species. Results obtained for CO<sub>2</sub> + O<sub>2</sub> mixtures confirm this view and suggest that back recombination processes involving CO and O<sub>2</sub> may reduce the overall splitting efficiency. The study of mixtures of CO<sub>2</sub> and dry air has proved the capacity of ferroelectric packed-bed reactors to efficiently decompose CO<sub>2</sub> with no formation of harmful N<sub>x</sub>O<sub>y</sub> subproducts in conditions close to those in real facilities. The found enhancement in energy efficiency with respect to that found for the pure gas decomposition supports that new reaction pathways involving nitrogen molecules are contributing to the dissociation reaction. We conclude that PZT moderated packed-bed plasma reactors is an optimum alternative for the decomposition of CO<sub>2</sub> in real gas flows and ambient conditions.

**Keywords:** Nonthermal plasmas, CO<sub>2</sub> decomposition, packed-bed reactor, atmospheric pressure plasma, ferroelectrics, optical emission spectroscopy (OES).

### References:

1. Navascues P., Cotrino J., González-Elípe A.R. Gómez-Rarmírez A. (2022) Plasma assisted CO<sub>2</sub> dissociation in pure and gas mixture streams with a ferroelectric packed-bed reactor in ambient conditions. *Chemical Engineering Journal* 430, 133066.

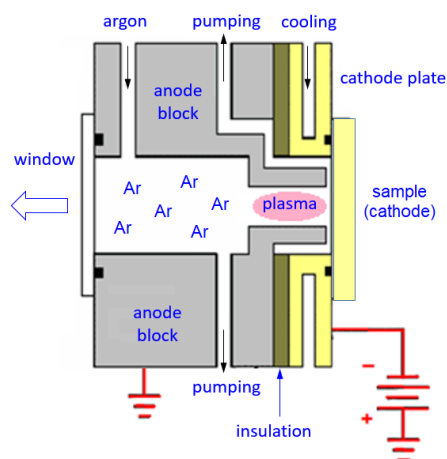
# Plasma Diagnostics in Glow Discharge Emission Spectroscopy: a Glimpse into the Excitation Processes Involved

Z. Weiss

FZU - Institute of Physics of the Czech Academy of Sciences, Praha, Czech Republic

## Abstract:

Glow discharge optical emission spectroscopy (GD-OES) with a Grimm-type source (Figure 1) is a well-established method for elemental analysis of metals, coatings and surface-modified materials. In most cases, a simple calibration-based procedure is sufficient to interpret analytically the emission intensities. In some situations, however, deviations from the calibration model occur and corrections are necessary. To establish such corrections, a good understanding of glow discharge excitation is desirable.



**Figure 1:** Grimm-type glow discharge excitation source: a flat sample to be analysed acts as a cathode in a glow discharge in argon. The spectrum is viewed end-on, in axial direction, and analyzed by an optical spectrometer.

Glow discharge excitation of several analyte elements has been studied experimentally using the high-resolution VUV-VIS Fourier transform spectrometer at Imperial College London, UK. Emission spectra, consisting typically of hundreds of lines of each analyte element were interpreted using two formalisms: (1) Experimental Boltzmann plots, expressing relative populations of excited levels of an atom or ion under study as function of their excitation energy, and (2) transition rate (TR) diagrams [1], expressing relative radiative depopulation rates of excited levels as function of their energy, and making thus possible to track individual decay paths of excited states, map cascade processes and describe thereby the excitation of the atom or ion under study. Major findings to be reported are as follows:

(1) Deviations from local thermodynamic equilibrium (LTE) exist and are caused by the fact that radiative decay of excited levels is not negligible compared to the deexcitation by electrons and violates thus microscopic reversibility in the excitation/deexcitation by electron collisions. The resulting Boltzmann plots are still close to linear but the thereby established ‘excitation temperatures’ of analyte atoms,  $T(\text{Me}^0)$ , and ions,  $T(\text{Me}^+)$ , are not equal, and follow the relation  $T(\text{Me}^0) < T(\text{Me}^+) < T_e$  where  $T_e$  is electron temperature and Me is the element under study [1].

(2)  $\text{Ar}^+ - \text{Me}^0$  asymmetric charge transfer (ACT) reactions cause selective over-population of the Me II (ionic) levels with total energies close to the argon ionization energy, 15.76 eV. Their decay enhances populations of lower Me II levels and this may affect the whole  $\text{Me}^+$  spectrum (=all other Me II levels that are excited from those low levels by electron or heavy particle collisions). The degree of ionization will then be higher than what would correspond to the equilibrium described by the Saha equation. If the Me II ground term or low metastable Me II levels are enhanced, this enhancement may also spread, due to electron collisions, into the highest Me I levels [1].

The effects mentioned were studied also in a hollow cathode discharge [2]. It was found that  $\text{Ar}^+ - \text{ACT}$  reactions are more strongly expressed in the flat-cathode configuration than hollow cathode.

**Keywords:** glow discharge, GD-OES, emission spectroscopy, Boltzmann plots, transition rate diagrams, LTE, local thermodynamic equilibrium, asymmetric charge transfer, ACT

## References:

1. Weiss, Z. (2020) Emission spectroscopic diagnostics of weakly ionized argon-diluted plasmas: glow discharge and inductively coupled plasma, *Plasma Sources Sci. Technol.*, 29, 105018
2. Weiss, Z., Concepcion-Mairey, F., Pickering, J.C., Smid, P. (2021) Emission spectroscopic study of an analytical glow discharge with plane and hollow cathodes: Titanium and iron in argon discharge, *Spectrochim. Acta Part B*, 180, 106208

# **Tribology 2022 Virtual Session**



## Graphene Coatings: A Disruptive Approach to Remarkable Corrosion Resistance

Raman Singh,

Department of Mechanical & Aerospace Engineering, Department of Chemical Engineering, Monash University, Australia

### Abstract

Corrosion and its mitigation costs dearly (any developed economy loses 3-4% of GDP due to corrosion, which translates to ~\$250b to annual loss USA). In spite of traditional approaches of corrosion mitigation (e.g., use of corrosion resistance alloys such as stainless steels and coatings), loss of infrastructure due to corrosion continues to be a vexing problem. So, it is technologically as well as commercially attractive to explore disruptive approaches for durable corrosion resistance.

Graphene has triggered unprecedented research excitement for its exceptional characteristics. The most relevant properties of graphene as corrosion resistance barrier are its remarkable chemical inertness, impermeability and toughness, i.e., the requirements of an ideal surface barrier coating for corrosion resistance. However, the extent of corrosion resistance has been found to vary considerably in different studies. The author's group has demonstrated an ultra-thin graphene coating to improve corrosion resistance of copper by two orders of magnitude in an aggressive chloride solution (i.e., similar to sea-water). In contrast, other reports suggest the graphene coating to actually enhance corrosion rate of copper, particularly during extended exposures. Authors group has investigated the reasons for such contrast in corrosion resistance due to graphene coating as reported by different researchers. On the basis of the findings, author's group has succeeded in demonstration of durable corrosion resistance as result of development of suitable graphene coating. The presentation will also assess the challenges in developing corrosion resistant graphene coating on most common engineering alloys, such as mild steel, and presents results demonstrating circumvention of these challenges.

# How to Slide (almost) without Friction from Boundary to Hydrodynamic Lubrication

J. Cayer-Barrio

Ecole Centrale de Lyon, Laboratoire de Tribologie et Dynamique des Systèmes, CNRS UMR5513, Ecully, France

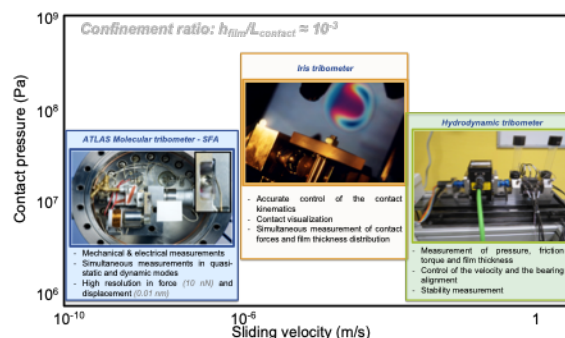
## Abstract:

Reducing friction dissipations is a promising way to contribute to the global welfare. To do so, the first approach is to use low-viscosity fluids and the second one is to modify the surfaces in contact.

We illustrate here the combined approach consisting in investigating the surface effects on friction in contacts lubricated with low-viscosity fluids, over all the lubrication regimes.

We will first present our new IMOTEP research platform that allows one to *in-situ* analyze the tribological mechanisms for conditions that cover 11 decades of sliding velocities and contact pressure, from  $10^{-10}$  to 10 m/s and up to few GPa [1-4], by combining simultaneous measurements of contact forces with film thickness distribution. We will second focus on an example, the tribological behavior of polymer solution. The interactions between the metallic surface and the polymer additive induced the formation of a 10-nm thick boundary layer, that was highly elastic, with a shear elastic modulus of 90 MPa under a 50MPa contact pressure. Interferogram analysis showed that patches of adsorbed film well covered the surface at low velocity. At high velocity, the film thickness followed the elastohydrodynamic prediction. In hydrodynamic lubrication regime, a micrometric hydrodynamic film separated the surfaces, highlighting the role of viscosity. The existence of this boundary film resulted in friction reduction, shifted the mixed/EHL regime transition towards lower velocities. In hydrodynamic regime, two regimes, thermal vs non-thermal, competed depending on the kinematics. The non-Newtonian fluid rheology under high shear was also investigated.

**Keywords:** friction reduction, surface, boundary lubrication, elastohydrodynamic lubrication, hydrodynamic lubrication.



**Figure 1:** *In-situ* multi-scale analysis of the lubrication regimes thanks to the IMOTEP research platform covering 11 decades of sliding velocities and contact pressure, from  $10^{-10}$  to 10 m/s and up to few GPa.

## References:

1. Crespo, A., et al. (2017), Methodology to Characterize Rheology, Surface Forces and Friction of Confined Liquids at the Molecular Scale Using the ATLAS Apparatus, *Tribology Letters*, 65, Article number 138.
2. Ernesto, A., Mazuyer, D., Cayer-Barrio, J. (2014), The Combined Role of Soot Aggregation and Surface Effect on the Friction of a Lubricated Contact, *Tribology Letters*, 55 (2014) 329–341.
3. Bonaventure, J., Cayer-Barrio, J., and Mazuyer, D. (2016), Transition Between Mixed Lubrication and Elastohydrodynamic Lubrication with Randomly Rough Surfaces, *Tribology Letters*, 64, 3, Article number 44.
4. Barazzutti, C., Cayer-Barrio, J., and Mazuyer, D. (2019), A new *in-situ* methodology for understanding hydrodynamics journal bearing, 8<sup>th</sup> International Tribology Conference, September 17 – 21, 2019, Sendai.

# Modeling of Lubrication between Parallel Rough Surfaces: Challenges and New Solutions

N. Brunetière<sup>1</sup>

<sup>1</sup> Department Institut Pprime, Dept GMSC, CNRS, University of Poitiers, Ensma, Poitiers, France

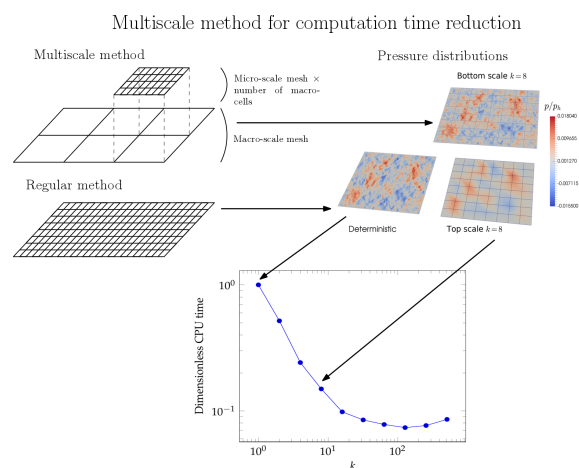
## Abstract:

In his pioneering work, Reynolds (1) was aware that roughness can affect the lubricant flow. First models considering roughness effect in thin film flows were proposed in the 60's. The 70's were probably the most fruitful period with the 4<sup>th</sup> Leeds Lyon Symposium fully dedicated to this topic and the famous papers of Patir and Cheng (2,3). They considered roughness effect as a perturbation of the solutions obtained with smooth surfaces. This method proves its efficient in many situations except for parallel rough surfaces. It is then necessary to used deterministic approaches based on very thin mesh capturing the detail of the surface topography. This alternate method is limited to small domains and by long computation times. Several multiscale methods (figure 1) can now push back these limitations (4,5). They will be briefly presented as well as their ability to evolve toward new hybrid and adaptive methods, the possible future methods for tribology simulation.

**Keywords:** Lubrication, roughness, mixed regime, multiscale methods, numerical simulation

## References:

1. Reynolds, O. (1886) On the Theory of Lubrication and Its Application to Mr. Beauchamp Tower's Experiments, Including an Experimental Determination of the Viscosity of Olive Oil, *Philosophical Transactions of the Royal Society of London*, 177, 157-234.
2. Patir, N., Cheng, H. (1978) An Average Flow Model for Determining Effects of Three-Dimensional Roughness on Partial Hydrodynamic Lubrication, *Journal of Lubrication Technology*, 100, 12-17.
3. Patir, N., Cheng, H. (1979) Application of Average Flow Model to Lubrication Between Rough Sliding Surfaces, *Journal of Lubrication Technology*, 101, 220-230.
4. Brunetiere, N., Wang, Q. (2014) Large-Scale Simulation of Fluid Flows for Lubrication of Rough Surfaces, *Journal of Tribology*, 136, 011701-14p.
5. Brunetière, N., Francisco, A. (2018) Multiscale Modeling Applied to the Hydrodynamic Lubrication of Rough Surfaces for Computation Time Reduction, *Lubricants*, 6, 83-12p.



**Figure 1:** Example of a multiscale finite element method used for lubrication simulation with rough surfaces (5).

# Study on the Effect of Water Content on Tribological Performance of Grease

S. Y. Chern, W. L. Liu, M. F. Gu

Department of Power Mechanical Engineering, National Formosa University

## Abstract:

Grease is an important type of lubricant for mechanical components. However, the lubricating performance of grease is often reduced due to the contamination of natural water content during the operation. This can lead to an earlier grease change and even machine downtime occurs. The effect of water content on mechanical parts is more significant, because the water content in grease will react with the surface of the parts to cause hydrogen embrittlement. When the surface material of the machine parts is damaged, the water content in the grease damages the mechanical parts more than particles affects the grease [1]. Grease life can be reduced by up to 90% with only 1% water content contained in the grease [2]. In this study, after simulating the contamination of water content in the grease, a four-ball wear test machine was used to conduct experiments. Then the wear scar diameters after experiments was measured by an optical microscope, and the absorbance of the grease before and after tests was measured by the LED red light optical module.

After the four-ball wear tests of the grease blended with 0.0, 0.5, 1.0 and 1.5 wt% water content, the results indicated that the more water content in the grease, the larger the wear scar diameter. Using LED red light to detect the absorbance of grease before and after tests, it is found that the red light absorbance, water content and wear have a positive relationship.

**Keywords:** Grease, Wear, Water Content, Absorbance.

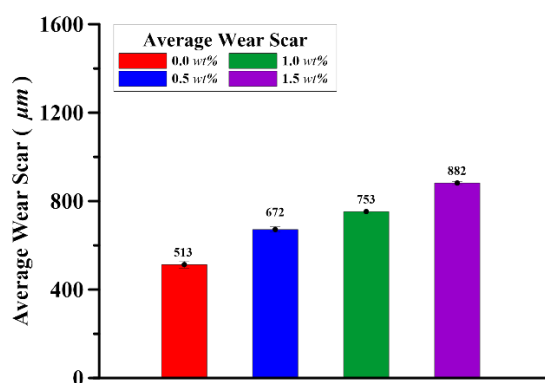


Figure 1 Comparison of the average wear scar

diameter after tests of grease with different water contents

**Figure 1** shown the change in wear scar diameter before and after the experiments of grease with different water contents. It is found that the more water content in the grease, the larger the wear scar diameter. Especially, the wear diameter is the largest at the water content of 1.5 wt% .

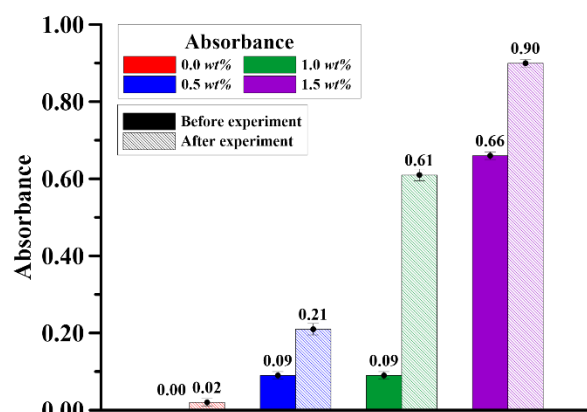


Figure 2 The absorbance of LED red light before and after tests of the grease with different water contents

**Figure 2** shows the change in the absorbance of LED red light before and after tests of the grease with different water contents. It is found that the absorbance of grease increases with the water content and increases faster when the water content is greater than 1.0 wt%. After the wear tests, the absorbance of grease also increases with the water content.

## References:

1. TOIMEN, "engineering manual timken ", pp. 48
2. D. Nicholas, 2015, "Mixing grease with water", Luleå tekniska universitet

# Influence of Laser Cladding on the Tribological and Microstructural Properties of Ex-Service Light Rails

P. Fasihi <sup>1\*</sup>, O. Kendall <sup>1</sup>, R. Abrahams <sup>1</sup>, C. Qiu <sup>2</sup>, P. Mutton <sup>2</sup>, W. Yan <sup>1</sup>

<sup>1</sup> Department of Mechanical and Aerospace Engineering, Monash University, Clayton VIC 3800, Australia

<sup>2</sup> Institute of Railway Technology, Monash University, Clayton, VIC 3800, Australia

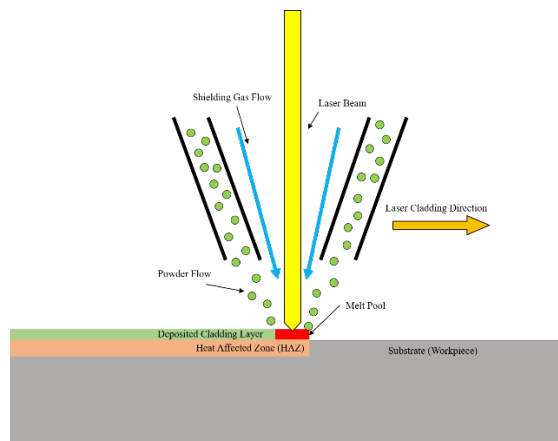
## Abstract:

Laser cladding is a surface engineering technique used to metallurgically bond a coating of metallic powder on a substrate (Figure 1) to improve its mechanical and tribological properties. The high-quality cladding layer enhance the wear and fatigue resistance as well as favourable microstructural characteristics which aims to protect the underlying bulk rail. Light rail is prone to rapid deterioration due to the low carbon steel grades commonly used in switch blades of tram networks. As these light rail systems are heavily relied upon for passenger transit, improving repair techniques to prolong the operation life is critical.

Laser cladding may be implemented as an additive manufacturing-based maintenance method to rebuild the profile of these worn components and deposit wear resistant coatings. In this study, laser cladding has been applied to rebuild worn, light rail components using Co-Cr based Stellite 6 and Stellite 21 alloys, and a new martensitic stainless steel. Tribological wear tests were performed on a custom-made roller on disc wear test machine. In comparison to the non-clad rails, the study demonstrated a noticeable decrease (about 93.9%) in the accumulated wear rate of the clad samples.

This study aims to show the suitability and benefits of laser cladding as an alternative maintenance strategy for light rail applications. Moreover, the findings from this study can be directly applied by railway infrastructure companies and industry operators to update and enhance their maintenance strategies.

**Keywords:** laser cladding, light rails, railway maintenance, tribological properties, microstructures.



**Figure 1:** Figure illustrating the schematic diagram of a laser cladding process [1].

## References:

1. Fasihi, P., Abrahams, R., Mutton, P., Yan, W. (2021), Tribological Properties of a New Alloy Laser Cladded on Hypereutectoid Rails, *J. Tribol.*, 143 (2021) 051110.

# Study on the Deterioration of Lubricating Oils with Environmental Particles

J. H. Horng, J. L. Lin, Y. Y. Lin

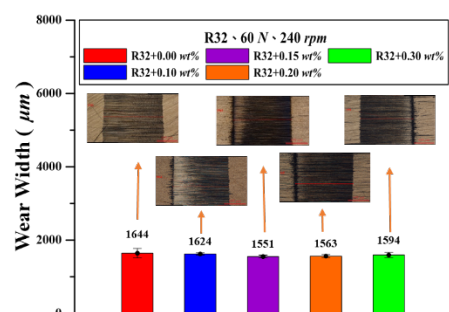
Department of Power Mechanical Engineering, National Formosa University, Yunlin, Taiwan (R.O.C.)

## Abstract:

Lubrication is an important technology to reduce friction and wear, and different lubricants are used for different component materials and operating conditions. Generally, lubricating oils at the contact interface between two components may be contaminated by environmental particles. In 2017, Liu et al. [1] found that the addition of silica nanoparticles in nanofluids show better anti-wear and anti-friction properties. Therefore, in this study different weight percentages of silica nanoparticles were added to lubricating oils having different viscosities (R32 and R68) to conduct experiments, and to simulate the situation that the lubricating oils is contaminated by foreign particles. A line contact tribotest machine was used to study the variation in tribological behaviors at the contact interface, and analyze the change in the characteristic of two oils blended with different weight percentage of the particles, then measure the deterioration degree of the lubricating oils.

The results indicated that the wear and oil temperature of both the R32 and R68 oils formulated with the silica nanoparticles show maximum values at the particle concentration of 0.10 wt%, and a minimum values at the particle concentration of 0.15 wt% under the same load and sliding speed, as shown in Figures 1 and 2. This tendency is the same as that in the research by Horng et al. [2].

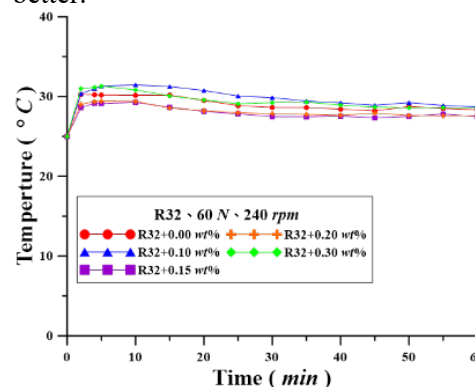
**Keywords:** Nanoparticle, Wear, Temperature.



**Figure 1** The wear scars after tests of the oil R32 with different concentrations of silica particles

**Figure 1** shows the change in wear scar diameters after the experiments with the oil R32

blended with non-concentrated silica particles. The best particles concentration was observed at 0.15 wt% while the worst concentration was at 0.10 wt%. Hence, adding an appropriate concentration of silica particles to the oil R32 can improve the anti-wear and anti-friction performance, but the concentration is not the more the better.



**Figure 2** The variation in temperature of the oil R32 added with different concentrations of silica particles

**Figure 2** shows the variation in oil temperature of the oil R32 formulated with different concentrations of silica particles during wear tests. The results show that appropriate addition of silica particles to the lubricating oil can reduce the oil temperature.

## References:

1. Liu X., Xu N., Li W., Zhang M., Chen L., Lou W., Wang X., (2017), Exploring the effect of nanoparticle size on the tribological properties of SiO<sub>2</sub>/polyalkylene glycol nanofluid under different lubrication conditions, *Tribology International*, Vol. 109, pp. 467-472.
2. Horng, J. H., Yu, C. C., Chen Y. Y., (2021), Tribological characteristics and load-sharing of point-contact interface in three-body mixed lubrication, *ASME, Journal of Tribology*. Vol. 144, 052201



# Defining a shear hardness: a computational approach

R. Capozza<sup>1\*</sup>, K. J. Hanley<sup>1</sup>

<sup>1</sup> School of Engineering, Institute for Infrastructure and Environment, The University of Edinburgh, Edinburgh EH9 3JL

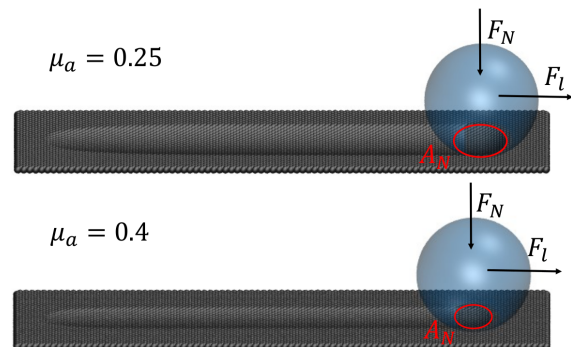
## Abstract:

Hardness is defined as the characteristic ability of a material to resist penetration or abrasion by other bodies [1]. Any quantitative measure of this feature of material behaviour depends on the technique used for its measurement. Several standard procedures have been developed for the interpretation of indentation hardness (Vickers, Brinell, Berkovitch). However, scratch hardness has not been formalized to the same extent.

Scratch hardness is the ability of one material to resist scratching or abrasion. It is often quantified through the concepts of “dynamic scratch hardness” or “ploughing hardness” [1, 2]. The former is defined as the normal force applied during a scratch test divided by the normal area,  $H_s = F_N/A_N$ ; the latter is the lateral force divided by the area orthogonal to the scratch direction,  $H_p = F_l/A_l$ . The resistance to scratching usually depends on the friction force at the interface and the dynamic scratch hardness clearly does not account for that. On the other hand, the ploughing hardness is practically difficult to use as the lateral area  $A_l$  can be negligibly small and difficult to quantify, especially in the case of a shallow scratch [3].

Here we introduce the new concept of “shear hardness” to characterize the resistance to scratching of a material. It is defined as the ratio of lateral force to normal area,  $H_{shear} = F_l/A_N$ . By using a novel approach for modelling abrasion, we give an operative definition of shear hardness. An immobile surface is discretized by a set of nodes and is abraded by a non-deformable particle. When the pressure on the surface overcomes a threshold, abrasion starts and the nodes are displaced along the normal to the surface to account for the surface change. From the consideration that a hard tip can scratch a softer surface even without an initial indentation, we show that the shear hardness is related to the indentation hardness through a coefficient  $\mu_a < 1$ , setting the effective resistance to scratching. As such a newly defined shear hardness is easily measured experimentally, it can lead to a new and efficient computational approach to model the surface abrasion of materials.

**Keywords:** surface hardness, computer simulations, friction, abrasion.



**Figure 1:** Scratching of a soft (top) and hard (bottom) material. The shear hardness is related to the indentation hardness through the coefficient  $\mu_a < 1$ , setting the effective resistance to scratching.

## References:

1. Williams, J., A. (1996) Analytical models of scratch hardness, *Tribology International*, 29, 675-694.
2. Leroch, S., Varga, M., Eder, S., J., Vernes, A., Rodriguez Ripoll, M., Ganzenmüller, G. (2011), Smooth particle hydrodynamics simulation of damage induced by a spherical indenter scratching a viscoplastic material, *International Journal of Solids and Structures*, 81 (2016) 188–202
3. Zhang, S., Guo, X., Jin, Z., Kang, R., Guo, D., Tang, W. C. (2020) Surface morphologies and corresponding hardness evolution during nanoscratching, *J mater res technol*, 9(3), 3179–3189

# Advancing Solid Interfaces and Lubricants by First principles Materials Design

M. Clelia Righi

Department of Physics and Astronomy “Augusto Righi”  
University of Bologna, Italy

## Abstract:

In this talk I will describe i) the workflow we developed for the high throughput screening of solid interfaces. A database for interfacial properties, such as adhesion and shear strength will be presented and discussed ii) the nanotribological properties of MXenes, focusing on the effect of surface terminations on interlayer and layer-substrate interactions i)

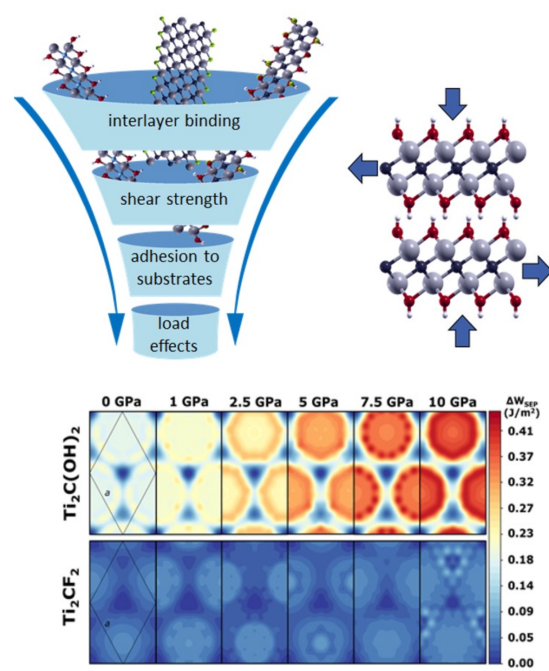
ii) First principles high throughput calculations have been successfully applied to screen the properties of hundreds of materials in an automated way. However, implementing a workflow for high-throughput calculations is challenging and requires robust IT infrastructures to collect, analyze, and save the data. To this end, we have designed and developed an advanced software package to study solid materials with density functional theory (DFT). It allows to carry out high-throughput analyses to evaluate the properties of bulks, surfaces, and interfaces. We used our package to calculate important tribological figures of merit connected to dry adhesive friction of solid-solid interfaces.

ii) MXenes are 2D transition metal carbides or nitride terminated with a mixture of  $-O$ ,  $-F$ ,  $-OH$  or  $-Cl$  surface terminations. The composition and structure of the layers enables the tuning of MXenes properties according to the target application. Here, we screen the effects of composition and termination on the tribological properties of MXenes. Our results indicate that interlayer adhesion and friction as well as the layer binding to iron and iron-oxide are governed by termination. Interestingly, MXenes with selective terminations show superior lubricating properties than MXenes with uncontrolled and mixed terminations.

**Keywords:** High throughput calculations, workflow, solid interfaces, adhesion and shear strength, MXenes

**Acknowledgments:** These results are part of the SLIDE project that has received funding from the European Research Council (ERC) under the European Union’s Horizon 2020 research and

innovation programme. (Grant Agreement No. 865633).



**Figure 1:** Figure illustrating the high throughput screening of MXenes’ tribological properties.

## References:

1. Jain, A. et al., “FireWorks: a dynamic workflow system designed for high-throughput applications.” *Concurrency Computat.: Pract. Exper.*, 27, 5037 (2015).
2. Restuccia, P. et al., “Ideal adhesive and shear strengths of solid interfaces: A high throughput ab initio approach” *Comp. Mat. Sci.* 154 517–529 (2018).
3. Wolloch, M. et al., “High-throughput screening of the static friction and ideal cleavage strength of solid interfaces” *Scientific reports*, 9(1), 1-10 (2019).
4. M Wolloch, M. et al “High-throughput generation of potential energy surfaces for solid interfaces” *Computational Materials Science* 207, 111302
5. G. Losi, O. Chehaimi and M. C. Righi, ”High throughput screening of the adhesion between surfaces of different materials”, in preparation.

6. Marquis, E. et al. "Impact of MXenes' Mixed Terminations on Interlayer Properties and Layer-Substrate Interaction", submitted.

# Detection and Prevention of Early Surface Degradation

Zaihao Tian<sup>1</sup>, Ping Lu<sup>1</sup>, Jo Grundy<sup>2</sup>, Shuncai Wang<sup>1</sup>, Robert Wood<sup>1,\*</sup>

<sup>1</sup>National Centre for Advanced Tribology at Southampton (nCATS), University of Southampton, Southampton, UK

<sup>1</sup>School of Electronics and Computer Science, University of Southampton, Southampton, UK

## Abstract:

Wear in highly stressed contacts can generate work function differences and charge distributions on wearing surfaces through phase transformations, material transfer and tribofilm evolution; thus detecting those early wear phenomena would inform the health of tribo-contacts and prevent catastrophic failure<sup>1,2</sup>. This talk investigates the early wear detection using multi-sensing techniques and machine learning.

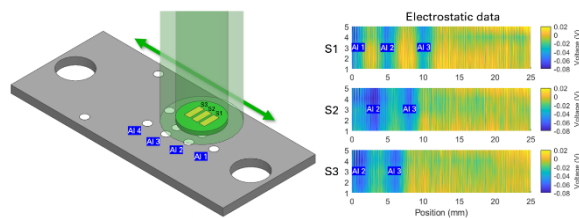
Calibration tests are reported of array electrostatic (ES) sensors using both a reciprocating facility and a twin rolling disc facility with different patterns of metals inserted. The calibration results show the sensors could distinguish metals and their pattern with different work functions (Figure 1). A 3D model was developed in COMSOL Multiphysics to simulate the ES array sensor response to charge patterns, and was found to be in good agreement with the calibration test results. Acoustic Emission (AE) sensors were used to detect elastic wave generation caused by asperity interactions and crack propagation and are compared and correlated to ES signals.

A twin-disc tribometer was then used to perform rolling contact fatigue experiments on bearing steel discs under rolling-sliding contacts in mixed lubrication regime. Optical and electron microscopes were used to identify wear patterns and measure wear development after initiation of tribofilms and pitting respectively. Features of early wear were extracted using time domain and frequency domain analyses of electrostatic and AE data. Time synchronous average analyses of electrostatic signals identified surface features which changed surface work functions i.e. tribofilms and pits. Time domain features of AE appeared to be sensitive to asperity contact conditions and frequency domain features were sensitive to crack propagation and pitting propagation. It is therefore concluded that electrostatic and AE provide considerable insight into early surface degradation.

Wear tests were also conducted on a reciprocating tribometer under starved lubrication conditions. The electrostatic signal was captured along with the stroke position and the friction force.

Five families of outlier detection algorithms were used in the data analysis, including regression, density mapping, ensemble, matrix and neural network methods. The outlier scores were calculated for each stroke, and successfully indicated the progression of wear on the surface.

**Keywords:** surface degradation, early wear, electrostatic sensing, acoustic emission sensing, condition monitoring.



**Figure 1:** Figure indicating the set-up and data results of electrostatic sensor testing. The electrostatic array sensor distinguishes different metals and identifies metal patterns.

## References:

1. Lu, P., Powrie, H.E., Wood, R.J., Harvey, T.J. and Harris, N.R., 2021. Early wear detection and its significance for condition monitoring. *Tribology International*, 159, p.106946.
2. Hutt, S., Clarke, A. and Evans, H.P., 2018. Generation of Acoustic Emission from the running-in and subsequent micropitting of a mixed-elastohydrodynamic contact. *Tribology International*, 119, pp.270-280.

# Triboscopy of Carbon Coatings Under Dry Friction Conditions

L. Lorenz <sup>1,2,\*</sup>, F. Härtwig <sup>1,2</sup>, S. Makowski <sup>2</sup>, M. Krause <sup>3</sup>, A. F. Lasagni <sup>1,2</sup>

<sup>1</sup> Institute of Manufacturing Science and Engineering, Technische Universität Dresden, Germany

<sup>2</sup> Fraunhofer Institute for Material and Beam Technology IWS, Dresden, Germany

<sup>3</sup> Helmholtz-Zentrum Dresden-Rossendorf, Dresden, Germany

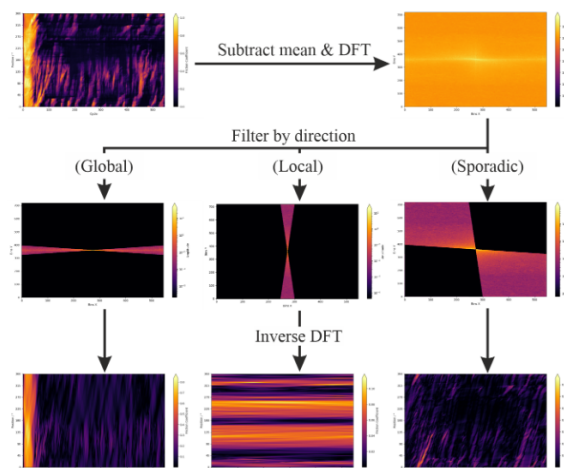
## Abstract:

Tetrahedral amorphous carbon (ta-C) has been shown to exhibit exceptionally low friction and wear under dry sliding in the presence of water vapor as well as acceptably low friction and wear in the presence of inert gases. It is known that these properties deteriorate rapidly under vacuum conditions with decreasing pressure [1].

For a better understanding of the wear mechanisms in doped and undoped ta-C coatings under vacuum and dry conditions, more advanced triboscopic imaging methods in regards to these systems were developed. Triboscopy has been described by Belin et al. as the numerical imaging of the evolution of a tribosystem and combines local and time-resolved information, most often the evolution of friction [2].

The experimental data shown in this study was obtained from a custom-built ultra high vacuum tribometer in ball-on-disc configuration. Based on these experiments, an extended classification to distinguish the different triboscopic features is suggested depending on the the persistence in both track position and time: Uniform, Global, Local, and Sporadic. Furthermore, a filter technique for quantifying triboscopic data in regards to this classification is introduced (Figure 1). The new and improved triboscopic techniques are applied to the various dry friction measurements of hydrogen-free carbon coatings under varying humidity and pressure. The resulting specific triboscopic features are correlated to distinct wear phenomena, demonstrating the increased analytical capabilities when compared to conventional friction curves and wear track images. While in the context of this work the correlations were made after the experiments, the use of these findings for monitoring in-line experiments can strongly enhance the capability of monitoring tribological systems in the future.

**Keywords:** triboscopic images, diamond like coatings, DLC, ta-C, a-C, ta-C:X, vacuum



**Figure 1:** Procedure for quantifying triboscopic data, by extracting the directional triboscopic features 2-D Fourier transform. The spatial information is well preserved and a quantitative evaluation is made possible.

## References:

1. J. Andersson, R.A. Erck, and A. Erdemir, Frictional behavior of diamondlike carbon films in vacuum and under varying water vapor pressure. *Surface and Coatings Technology* 2003, 163-164, 535–540.
2. M. Belin and J.M. Martin, Triboscopy, a new approach to surface degradations of thin films, *Wear*, 156 (1992) 151-160.

## Acknowledgment:

This work was supported by the Deutsche Forschungsgemeinschaft (DFG – German Research Foundation) under grant agreement 415726702.



# Hydrostatic load on wavy sealing surfaces

De Huang<sup>1\*</sup>, Xiang Yan<sup>2</sup>, Roland Larsson<sup>1</sup> and Andreas Almqvist<sup>1</sup>

<sup>1</sup> Division of Machine Elements, Luleå University of Technology, Luleå 97187, Sweden.

<sup>2</sup> Saint-Gobain Seals, Bristol, RI 02809, USA.

## Abstract:

Fluid between contacting surfaces is inevitable due to the non-conform contact. The interfacial fluid between the contacting surfaces can exist in several states. Before the percolation threshold, there are flow paths that penetrate the contacting surfaces from inlet to outlet. Meanwhile, there is also fluid being blocked halfway and never able to reach the outlet. Moreover, the fluid can be surrounded by contacting areas and isolated from both the flow inlet and outlet. It is of interest to understand the effect of interfacial fluid on the contact state of the sealing surfaces and the sealing performance. Fluid-Structure Interaction (FSI) need to be considered when studying the sealing problem with fluid between interface [1]. In the current study, A finite element model is built to simulate a wavy sealing surface with fluid both isolated from and connect to the high-pressure side. The main research interest is how the contact state change with varying loading conditions and the effect of the initial trapped volume of the fluid between contacting interfaces.

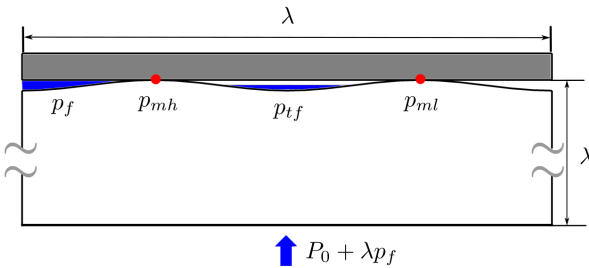


Figure 1: Problem set-up for the FEM model. A linear elastic wavy surface  $y(x) = \Delta \cos(4\pi x/\lambda)$  of length  $\lambda$  contact with a flat rigid body under a total vertical load  $P_0 + \lambda p_f$ , where  $P_0$  is the line load density of the pre-tension and  $p_f$  is the sealed fluid pressure. The trapped fluid pressure  $p_{tf}$  is determined through equation (1). The two peak contact pressure are  $p_{mh}$  and  $p_{ml}$ .

A plane strain problem is studied for an assembly shown in Figure 1. The fluid compressibility is considered for the trapped fluid and the magnitude of trapped fluid pressure  $p_{tf}$  is determined by [2]:

$$p_{tf} = p_f^* ((V/V_i)^{(-k)} - 1), \quad (1)$$

with  $V_i = \alpha V_0$ , where  $V_0$  is the closed gap volume between the contacting surfaces without loading and  $\alpha$  is the initial volume ratio of the trapped fluid in the closed gap. The parameters  $p_f^*$  and  $k$  is

determined by fluid properties.

We observed that the trapped fluid pressure  $p_{tf}$  increases with sealed fluid pressure  $p_f$  as it is compressed, and the increase rate of  $p_{tf}$  slows down with higher  $p_f$ . The trapped fluid pressure can approach to the sealed fluid pressure but can never be greater than  $p_f$  in the current study, as shown in Figure 2(a). The sealed fluid between contacting surfaces generates a bending moment on the seal. To counter this moment, more contact pressure shifts to the low-pressure side of the seal and create higher peak contact pressure at the low-pressure end. This explains that the peak contact pressure ratio first increases as  $p_f$  increases.

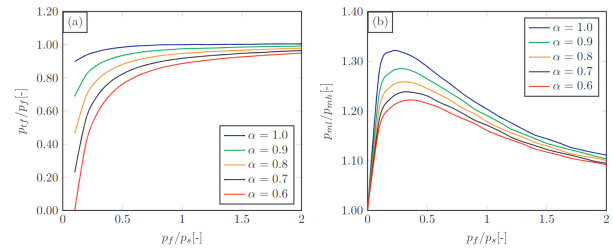


Figure 2: (a) The relationship of trapped fluid pressure  $p_{tf}$  with sealed fluid pressure  $p_f$ , for different initial volume ratio of the trapped fluid  $\alpha$ . (b) The relationship between the peak contact pressure ratio  $p_{ml}/p_{mh}$  and sealed fluid pressure  $p_f$ , for different initial volume ratio of the trapped fluid  $\alpha$ . The pressure is scaled with  $p_s = \frac{2\pi E \Delta}{(1-\nu^2)\lambda}$ , where  $E$  is elastic modulus and  $\nu$  is the Poisson ratio. The pre-tension  $P_0 = 0.014\lambda p_s$ .

**Keywords:** Seal, Contact mechanics, Trapped fluid.

## References

1. A. Shvarts, "Coupling mechanical frictional contact with interfacial fluid flow at small and large scales," PhD Thesis, 2019.
2. Y. A. Kuznetsov, "Effect of fluid lubricant on the contact characteristics of rough elastic bodies in compression," *Wear*, vol. 102, no. 3, pp. 177–194, Apr. 1985, doi: 10.1016/0043-1648(85)90217-0.



# Laser-based processing of polymeric tribological coatings for light-weight applications

M. Dahmen <sup>1</sup>, C. Vedder <sup>1</sup>, J. Stollenwerk <sup>1,2</sup>

<sup>1</sup> Fraunhofer Institute for Laser Technology (ILT), Steinbachstraße 15, 52074 Aachen, Germany

<sup>2</sup> Chair for Technology of Optical Systems TOS, RWTH Aachen University, Steinbachstraße 15, 52074 Aachen, Germany

## Abstract:

Automotive and aerospace engineering is characterized by an ever-increasing reduction in energy consumption, partly due to restrictions imposed by government regulations. This leads to new innovations for reducing friction and wear in light-weight components. In particular, components that are in constant frictional contact have a high potential for further improvements in tribological properties. High-performance polymers like polyether ether ketone (PEEK) are potential candidates to substitute state of the art friction-reducing coatings due to their excellent properties in terms of temperature resistance, friction reduction as well as corrosion and wear protection.

Conventionally, PEEK is applied to the component in a microparticulate dispersion. It is then melted in an oven-based process, whereby the entire component is heated above the melting temperature of PEEK ( $T_M = 340\text{ °C}$ ) for minutes up to hours [1]. Due to the high thermal load on the component, temperature-sensitive materials cannot be processed without a significant reduction in the hardness of the component. Engine pistons are usually made of an aluminum alloy, which is why it is not possible to apply these coatings using conventional methods.

An innovative approach to overcome this deficit is to form a dense and adhesive protection layer by melting the microparticulate PEEK layer using laser radiation. Due to the rapid heating and cooling rates, laser radiation offers the possibility to achieve the necessary melting temperature within the coating while reducing the thermal load on the substrate material. This leads to new challenges in obtaining sufficient adhesion between coating and substrate due to the comparably low substrate surface temperature during the process [2,3].

The present study deals with the development of a laser-based melting process using two laser beam sources to produce adhesive and dense PEEK coatings for tribological applications. Aditivation of the powder makes it possible to further enhance the performance of the coating in terms of corrosion protection, wear protection

and lubricant film formation. The individual layers can be applied one on top of the other by means of printing or spraying processes and subsequent laser post-treatment, allowing discrete multi-layer systems with application-adapted properties to be produced.

**Keywords:** Laser-based processing, Laser melting, High-performance polymers, Polyether ether ketone, PEEK, Tribological coatings, Surface treatments, Coating application, Temperature sensitive materials



**Figure 1:** Figure illustrating a polymeric tribological coating on an engine piston produced in a laser-based process.

## References:

1. Victrex Polymer Solutions. (2018) Vicote™ Coating 700 Series, Quick 10 Step Guide For Optimum Results. Retrieved from [https://www.victrex.com/~media/literature/en/victrex\\_vicote-700-series-10-step-us.pdf](https://www.victrex.com/~media/literature/en/victrex_vicote-700-series-10-step-us.pdf)
2. Sändker, H., Stollenwerk, J., et al. (2017) Laser-based process for polymeric tribological coatings on light weight components, *Surface & Coatings Technology*, 332, 391-398.
3. Sändker, H. (2018) Laser-based Production of Functional Coatings Made from Particulate Polyetheretherketone [Doctoral dissertation]. RWTH Aachen University.

# Weighted LASSO feature selection for the analysis of FT-IR spectra applied to relate engine oil degradation patterns

Pia Pfeiffer<sup>1</sup>, Bettina Ronai<sup>2</sup>, Georg Vorlaufer<sup>2</sup>, Nicole Dörr<sup>2</sup>, Peter Filzmoser<sup>1</sup>

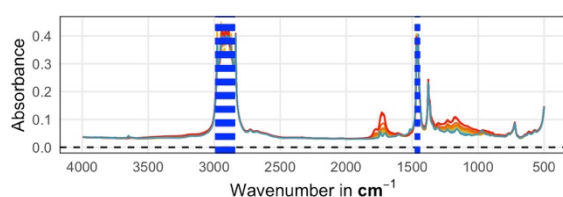
<sup>1</sup> Institute of Statistics and Mathematical Methods in Economics, TU Wien, Wiedner Hauptstraße 8-10, 1040 Vienna, Austria

<sup>2</sup> AC2T research GmbH, Viktor-Kaplan-Straße 2/C, 2700 Wiener Neustadt, Austria

## Abstract:

FT-IR spectroscopy is a popular and widely applied method for the analysis of fresh and degraded lubricants that has also been used to predict oil attributes such as Viscosity Index (VI), Total Acid Number (TAN) or Total Base Number (TBN) and oil degradation [1]. Recent advances in artificial oil alteration allow for the production of large quantities of degraded oils under laboratory-controlled conditions [2]. To adjust a defined degree of degradation qualitatively and quantitatively in the laboratory, knowledge of the degradation mechanisms in the field and their connection with the parameters of artificial alteration is indispensable. Using weighted LASSO regression [3], this work contributes to understanding the quantitative relationship between artificial alteration and degradation in field use.

A comprehensive data analysis pipeline is presented and demonstrated on a real-world dataset consisting of FT-IR spectra of in total 58 engine oils in different conditions: First, a PCA-reconstruction-error based procedure to filter non-informative variables is presented. This preprocessing method does not rely on manual selection of the FT-IR absorption bands that are suitable for analysis, but performs the filtering of non-informative variables objectively. Figure 1 illustrates the result of the filtering method.

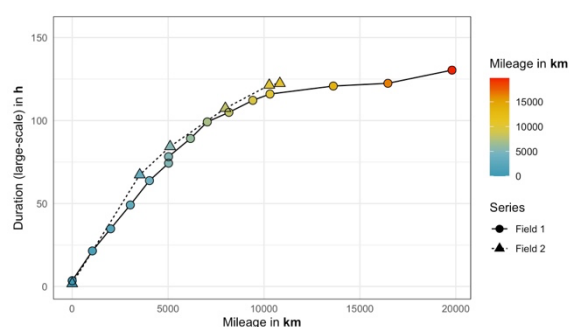


**Figure 1.** Diagnostic plot for filtering: The blue-dashed ranges are identified as non-informative.

Then, (weighted) LASSO regression, motivated both from a mathematical and chemometric point of view, is applied to the given dataset. Furthermore, suitable statistical methods to derive confidence intervals based on the concept of post-selection inference [4] and measures for variable importance are discussed. Using weighted LASSO, experts' knowledge can be integrated with model building, and variable selection is performed during the estimation process. This

leads to simple models with very few variables and excellent prediction performance.

The presented methods are applied to model how different patterns of artificial oil alteration as well as field use are associated. Figure 2 visualizes the relation between artificial large-scale alteration and field use according to our model.



**Figure 2.** The model leads to high predictive power between duration in artificial large-scale alteration and mileage in field use.

**Keywords:** oil condition monitoring, chemometrics, spectroscopy, high-dimensional data analysis

## References:

1. Felkel, Y., Dörr, N., Glatz, F., Varmuza, K., 2010. Determination of the total acid number (TAN) of used gas engine oils by IR and chemometrics applying a combined strategy for variable selection. *Chemometrics and Intelligent Laboratory Systems* 101, 14–22
2. Agocs, A., Budnyk, S., Besser, C., Ristic, A., Frauscher, M., Ronai, B., Dörr, N., 2020. Production of used engine oils with defined degree of degradation in a large-scale device. *Acta Technica Jaurinensis* 13, 131–150.
3. Hastie, T., Tibshirani, R., Wainwright, M., 2015. *Statistical Learning with Sparsity: The Lasso and Generalizations*. 1st ed., Chapman and Hall/CRC.
4. Lee, J.D., Sun, D.L., Sun, Y., Taylor, J.E., 2016. Exact post-selection inference, with application to the lasso. *Annals of Statistics* 44, 907–927.

# Analysis of the incomplete film in journal bearings using computational fluid dynamic

S. Wei , Y. Kligerman, R. Goltsberg\* and I. Etsion  
Department of Mechanical Engineering, Technion, Haifa, Israel

## Abstract:

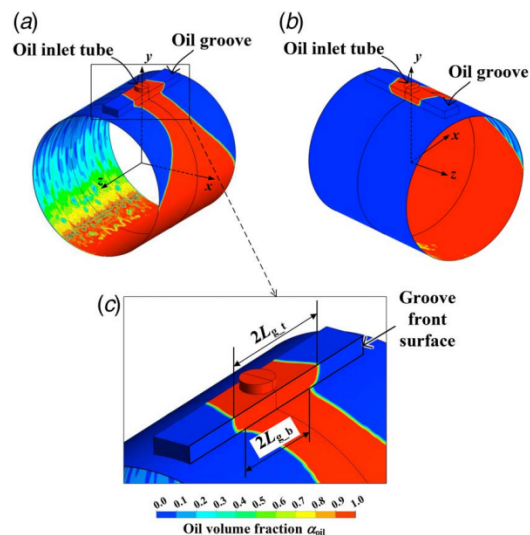
An axial groove is a common lubricant supply method for hydrodynamic bearings to distribute lubricant over the axial length of the bearings. Full starting lubricant film has become a common assumption in the theory of hydrodynamic lubrication when solving the Reynolds equation. However, the theoretical solution with this assumption yields a poor correlation to experimental observations when side leakage is considered. To resolve this issue an incomplete film upstream boundary conditions should be adopted. The length of the starting film should depend on the lubricant supply conditions such as supply pressure, load, and inlet geometry.

To clarify the realistic upstream boundary conditions of hydrodynamic bearings, a computational fluid dynamics analysis of two-phase (due to cavitation) flow was used to obtain the distribution of lubricant in a journal bearing, including inlet tube and groove.

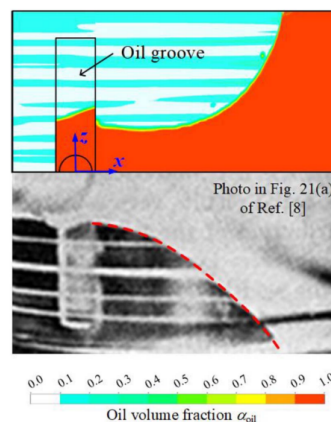
By intensive parametric study for clearance, velocity, and inlet mass flow rate, an empirical expression for the oil spread-length is derived for a simplified geometry of parallel plates [1] and for the complex geometry of a journal bearing [2].

It was found that for an incomplete starting film, the oil spread-length varies along the groove depth and film thickness (Figure 1). The numerical simulations also show that in the cavitation region, the streamlets do not fill the entire film thickness. A comparison with experimental observation (Figure 2) validates the present model as a proper one for accurately solving oil spread in similar systems.

**Keywords:** lubricant supply method, incomplete lubricant film, journal bearing, upstream boundary conditions, CFD analysis, bearing design and technology, fluid film lubrication, lubricant distribution, streamlets.



**Figure 1:** Distribution of  $\alpha_{oil}$  on (a) front view and (b) back view of the entire journal bearing and (c) zoom in on the groove region. Taken from [2].



**Figure 2:** Comparison of oil spread and streamlets between the CFD simulation and the experimental observation. Taken from [2].

## References:

1. Wei, S., Zhang, H., Kligerman, Y., Goltsberg, R., and Etsion, I. (February 23, 2021). "Analysis of Incomplete Film in Parallel Plates Including Inlet Tube and Groove." ASME. *J. Tribol.* November 2021; 143(11): 111804. <https://doi.org/10.1115/1.4050016>.
2. Wei, S., Kligerman, Y., Goltsberg, R., and Etsion, I. (December 8, 2021). "Variation of Lubricant Distribution Across the Radial Direction in a Journal Bearing." ASME. *J.*

*Tribol.* June 2022; 144(6):  
061803. <https://doi.org/10.1115/1.4053093>

# Unexpected low friction property of brass on ta-C in vacuum and atmospheric conditions

Fabian Härtwig<sup>1,2</sup>, Lars Lorenz<sup>1,2</sup>, Stefan Makowski<sup>2</sup>, Matthias Krause<sup>3</sup>, Carsten Habenicht<sup>3</sup> and Andrés Fabián Lasagni<sup>1,2</sup>

<sup>1</sup> Institute of Manufacturing Science and Engineering, Technische Universität Dresden, Germany

<sup>2</sup> Fraunhofer Institute for Material and Beam Technology IWS, Dresden, Germany

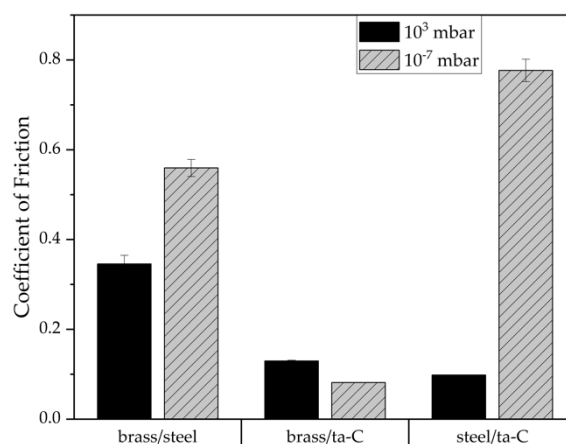
<sup>3</sup> Helmholtz-Zentrum Dresden-Rossendorf, Dresden, Germany

## Abstract:

It is well known that tribological systems operating in vacuum environments present severe challenges to be overcome. MoS<sub>2</sub> is one of the most well-known materials for providing low friction in both ambient and vacuum environments. However, it has a low wear resistance and a limited ability to withstand higher contact pressures. Super-hard hydrogen-free tetrahedral amorphous carbon coatings (ta-C) are a possible wear-resistant alternative. While they have low friction and wear properties in air at normal atmospheric conditions, their vacuum performance is limited when self-paired or in combination with steel.

In this work, the impact of various counter body materials is studied when paired with ta-C coatings. The evaluated counter body materials include brass, bronze, copper, silicon carbide, as well as aluminum oxide (Al<sub>2</sub>O<sub>3</sub>), steel and ta-C coated. In particular, brass was found to be the most promising counterbody material, and it was put to the test in direct comparison to steel as well as in long-term performance tests. In the short term, regardless of pressure, the brass/ta-C friction pair exhibits low friction (~0.1) and high wear, whereas in the long term, the friction coefficient increases (up to 0.52) due to a change in wear mechanism. Al<sub>2</sub>O<sub>3</sub> was found to be a promising sliding partner against ta-C, with a higher friction coefficient (0.3) but significantly lower wear than brass.

**Keywords:** diamond like coatings, DLC, ta-C, tribology, vacuum



**Figure 1:** Steady-state friction coefficients of tribological experiments performed with the tribo pairs brass/steel, brass/ta-C and steel/ta-C pairs, at 10<sup>3</sup> mbar and 10<sup>-7</sup> mbar.

## Acknowledgment:

This work was supported by the Deutsche Forschungsgemeinschaft (DFG – German Research Foundation) under grant agreement 415726702 and Sächsische Aufbaubank (SAB) within project LUBRICOAT.

# **Plasma Tech 2022 Virtual Session**



## Aerosol assisted plasma deposition of composite coatings: from biomaterials to catalysis

F. Palumbo<sup>1,\*</sup>, C. Lo Porto<sup>2</sup>, P. Favia<sup>1,3</sup>

<sup>1</sup> Insitute of Nanotechnology, CNR, Bari, Italy

<sup>2</sup> Insitute of Chemical and Physical Processes, CNR, Bari, Italy

<sup>3</sup> Department of Chemistry, University of Bari Aldo Moro, Bari, Italy

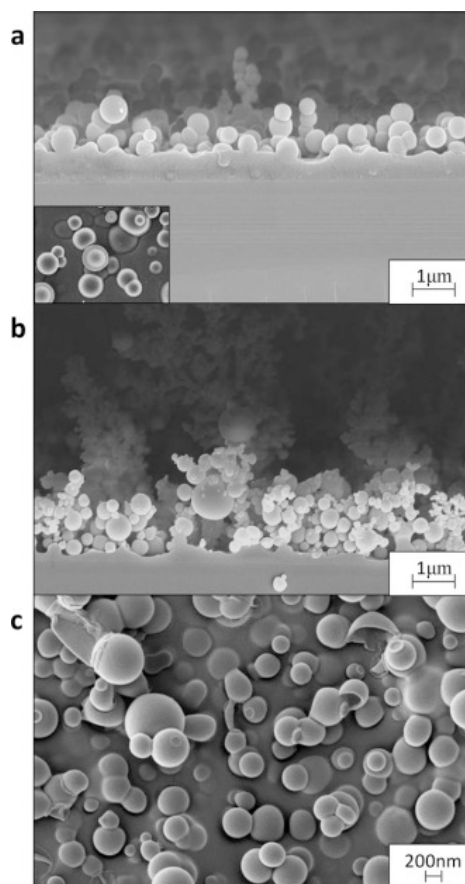
### Abstract:

Atmospheric pressure plasma is a versatile tool for the modification of materials surfaces with a wide range of applications. Aerosol injection of precursors can be coupled with atmospheric pressure plasma largely extending the flexibility of this kind of process. In fact both solid and scarcely volatile precursors can be delivered to the plasma, increasing the assortment of chemical surface modification. In the case of solid compound a suitable solvent is chosen for its dispersion. Furthermore, labile bio-active molecules, that commonly can be damaged in the reactive plasma environment, when injected with aerosol are less prone to chemical alteration, since they are protected by a solvent shell.

In this conditions, the deposition of drug containing coatings can be easily achieved, with application in the biomedical field as drug delivering systems, biosensing device and alike. Gentamicin, vancomycin and lysozyme containing films have been deposited preserving the antimicrobial activity. Such coatings show a release kinetic when immersed in water. Furthermore, it will be demonstrated that with a proper choice of aerosol solution and addition of ethylene as a source of the embedding matrix, unique core-shell nanocapsules can be obtained, as reported in fig 1.

On the other hand it will be shown that photocatalytic nanoparticles can be injected in plasma giving origin to nanocomposite coatings useful for water pollutants degradation.

**Keywords:** nanocomposite, antibacterial, plasma deposition, aerosol



**Figure 1:** Cross-sectional SEM images of plasma-deposited coatings (20 sccm ethylene, 5 slm He, 15 mg/ml of vancomycin solution) in (a) continuous mode, (inset top view) and (b) pulse mode condition. (c) Top view: SEM image of a CM plasma-deposited coating after 60 min immersion in water.

# Correlation of Excited Chemical Species by Solution Plasma in Cyclic Organic Compounds and the synthesized Carbon-based structure

Jiangqi Niu<sup>1</sup>, Chayanaphat Chokradjaroen<sup>1</sup>, Nagahiro Saito<sup>1,2,3,4,\*</sup>

<sup>1</sup> Department of Chemical Systems Engineering, Graduate School of Engineering, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

<sup>2</sup> Conjoint Research Laboratory in Nagoya University, Shinshu University, Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

<sup>3</sup> Japan Science and Technology Agency (JST), Open Innovation Platform with Enterprises, Research Institute and Academia (OPERA), Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

<sup>4</sup> Japan Science and Technology Agency (JST), Strategic International Collaborative Research Program (SICORP), Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

\* hiro@sp.material.nagoya-u.ac.jp

## Abstract:

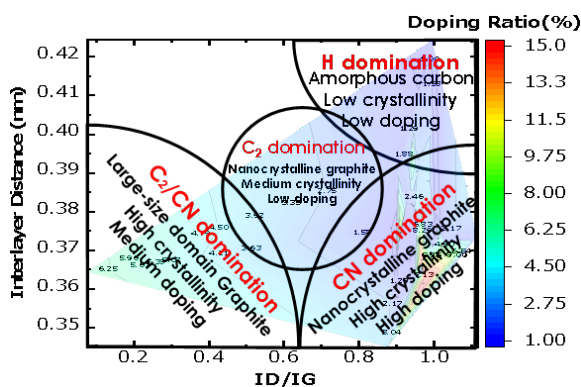
Carbon-based rechargeable lithium-ion batteries have been intensively commercialized since the development by Sony Laboratory. Lithium is replaced by a carbon host structure that can reversibly absorb and release lithium ions at low electrochemical potentials. Furthermore, the N-doping of the carbon material shows a significant increase in specific capacity relative to the pure carbon structure, enhancing the performance of the lithium battery. It is worth noting that solution plasma is a simple method for synthesizing high proportions of N-doped carbon. However, to date, there is no effective condition for the stable synthesis of the desired N-doped carbon. This is because there are numerous parameters which include equipment-dependent parameters and physical property parameters in the solution plasma process. And they have complex relationships with each other.

In solution discharge, the carbon formation reaction is occurred by undergoing electron transfer of plasma and molecules. In the plasma reaction field, gaseous atoms and molecules are excited to different levels under conditions of inelastic collisions of free electrons, resulting in excited chemical species. Different atoms or molecules radiate in a specific wavelength range, which corresponds to the potential energy difference between the upper excited state and the lower layer. This fine wavelength component can be captured using a high-resolution optical emission spectrometer and is presented as a peak profile. It provides information about the emitted species in the solution plasma reaction field, however, these emitted species are the fundamental processes that form the carbon structures.

As the result shown in Figure 2, there is a correlation between the peak profiles obtained by emission analysis in the plasma and the final

carbon products. If the emission profile is C<sub>2</sub> or CN domination, it will induce the synthesis of graphite products with a larger size, high crystallinity, and around 7 at % dopants. This result shows that the emission profile and index of excited chemical species could act as an index to indicate the synthesis of different carbon structures in the case of solution plasma reaction field for cyclic organic compounds to carbon.

**Keywords:** Solution plasma, excited chemical species, optical emission profiles, N-doped carbon, correlation.



**Figure 1:** Correlation mapping of Excited Chemical Species Produced by Solution Plasma in Cyclic Organic Compounds and the final carbon products.

## References:

1. Morishita, T.; Ueno, T.; Panomsuwan, G.; Hieda, J.; Yoshida, A.; Bratescu, M. A.; Saito, N. (2016), Fastest Formation Routes of Nanocarbons in Solution Plasma Processes, *Sci. Rep.*, 6, 36880.

# Reduction Synthesis of High-Entropy Alloy Nanoparticles in the Plasma Ionic Liquid System and their Application to Hydrogen Evolution Reaction

G. Lee<sup>1</sup>, V.-T. Nguyen<sup>1</sup>, L. Larina<sup>1</sup>, N.-A. Nguyen<sup>1</sup>, H.-S. Choi<sup>1,2\*</sup>, M. Keidar<sup>2</sup>

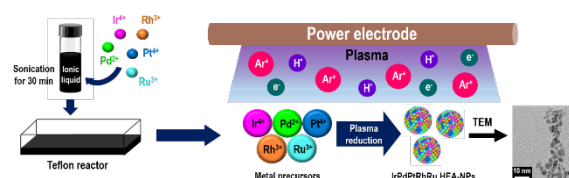
<sup>1</sup> Department of Chemical Engineering and Applied Chemistry, Chungnam National University, Daejeon 34134, Korea

<sup>2</sup> Department of Mechanical and Aerospace Engineering, The George Washington University, Washington DC 20052, USA

## Abstract:

Unlike the conventional alloy composed of two or not more than three elements, the high entropy alloy (HEA) is defined as a single-phase solid solutions in which at least five dominant elements are present.<sup>1</sup> Each element ranges from 5 to 35 atomic percentage without forming fragile inter-metallic compounds.<sup>2,3</sup> Herein, we report a novel cost effective and scaling process for syntheses of the quinary IrPdPtRhRu HEA-NPs (Figure 1). The process is based on the plasma ionic liquid reduction strategy.<sup>4</sup> The combination of PXRD, HRTEM, XPS and EDX analyses confirm that application of the developed wet plasma reduction method yields the IrPdPtRhRu HEA-NPs. HEA-NPs with size of  $\approx 5$  nm were synthesized under mild temperature, atmospheric pressure, and without the support assistance. All elements constituting the as-prepared IrPdPtRhRu HEA-NPs are uniformly distributed in the FCC single-phase nanostructure. The valence band emission suggests the hybridization of the metal orbitals in the IrPdPtRhRu HEA-NPs. The work function (WF) of 4.63 eV for the HEA is determined by UPS. WF value is lower than WFs of the metals from which the HEA consists, suggesting the higher catalytic activity. The IrPdPtRhRu/C electrocatalyst shows excellent catalytic performance toward hydrogen evolution reaction (HER) with the overpotential of 60 mV at a current density of  $10 \text{ mA cm}^{-2}$ . Tafel slope of  $40 \text{ mV dec}^{-1}$  was recorded in the alkaline electrolyte. HEA electrocatalyst exhibits a long-term stability for 6 h without significant decay under a high constant current density of  $100 \text{ mA cm}^{-2}$ . The findings in this study contribute to the basic science understanding of catalysts and provide a platform for further development of HEA-NPs electrocatalysts for large-scale applications.

**Keywords:** high-entropy alloy nanoparticles, wet-chemistry method, plasma-ionic liquid reduction, electrocatalyst, hydrogen evolution reaction.



**Figure 1:** Schematic illustration of synthesis process of IrPdPtRhRu HEA-NPs using wet plasma reduction method.

## References:

1. Ye, J.W., Wang, Q., Lu, J., Liu, C.T., Yang, Y., (2016) High-entropy alloy: challenges and prospects, *Mater. Today* 19, 349-362.
2. Miracle, D.B., Senkov, O.N., (2017) A critical review of high entropy alloys and related concepts, *Acta Mater.* 122, 448-511.
3. Tsai, M.H., Li, J.H., Fan, A.C., Tsai, P.H., (2017) Incorrect predictions of simple solid solution high entropy alloys: Cause and possible solution, *Scr. Mater.* 127, 6-9.
4. Tran, Q.C., Dao, V.D., Kim, H.Y., Jung, K.D., Choi, H.S., (2017) Pt-based alloy/carbon black nanohybrid covered with ionic liquid supramolecules as an efficient catalyst for oxygen reduction reactions, *Appl. Catal. B* 204, 365-373.

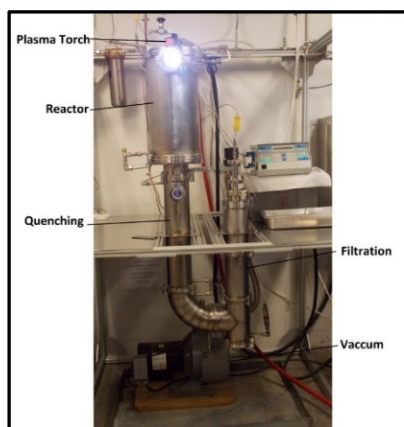
# Synthesis of SiC Nanopowder by DC Plasma for Nuclear Fuel Applications

G. Cota-Sanchez<sup>1</sup>, D. McDonald<sup>1</sup>, J. Mouris<sup>1</sup>, N. Lee<sup>1</sup>

<sup>1</sup> Canadian Nuclear Laboratories, Chalk River, Ontario, Canada

## Abstract:

Silicon carbide (SiC) is being widely investigated in the nuclear industry for a number of advanced fuel applications [1], [2]. Canadian Nuclear Laboratories (CNL) is conducting research and development on enhanced accident-tolerant fuels (ATF) for the Canadian Supercritical Water Reactor (SCWR) through its Federal, Science and Technology-Energy program. Typical SiC applications include, as coating material for cladding applications [1] and as matrix material in TRISO-structural ISOtopic (TRISO) fuel fabrication [2]. A plasma-based method is being developed for the synthesis of SiC nanopowder. The method is based on the use of a 40-kW DC thermal plasma torch for decomposition of silicon-organic materials and formation of SiC nanoparticles. The experimental system comprises mainly three sections [3]. The first section includes the plasma torch and reactor used to decompose the raw material. The second section includes the quenching zone where SiC condenses and the third section includes the filtration system where SiC nanoparticles are collected. Figure 1 depicts the experimental set-up used in the tests.

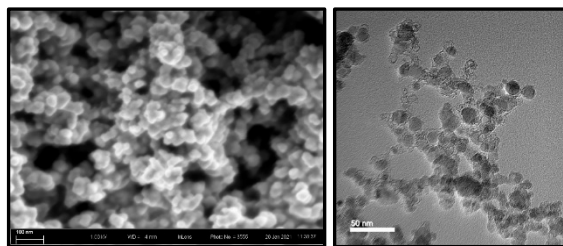


**Figure 1:** Experimental Plasma System [3]

Theoretical work included equilibrium thermodynamic studies performed to estimate SiC, carbon (C), and silicon oxide (SiO<sub>2</sub>) stability zones at plasma conditions. The study showed that solid SiC is stable at temperatures above 500 K.

Experimental work consisted of several tests performed to evaluate the effect of electrical power,

reactor pressure, plasma gas, and quenching rates on the production of SiC. Figure 2 shows SEM and TEM images of the produced SiC nanopowder. A particle size of less than 50 nm was estimated using SEM and TEM images.



**Figure 2:** Synthesized SiC Nanopowder. SEM (Left) and TEM (Right) Images

SiC nanoparticles were successfully produced using a methodology based on a thermal plasma system. Both theoretical and experimental studies showed that C, and SiO<sub>2</sub> generation can be prevented by optimizing the operating conditions of the plasma process, enhancing the production of SiC.

**Keywords:** DC thermal plasma, silicon carbide, nanoparticles, thermodynamic equilibrium, nuclear applications.

## References:

1. Barrett, K., Bragg-Sitton, S., Galicki, D., (2012). *Advanced LWR Nuclear Fuel Cladding System Development Trade-off Study*, INL-EXT-12-27090.
2. Sawa, K., (2012). *TRISO Fuel Production*. Comprehensive Nucl. Mat. 3, 143-149.
3. Cota-Sanchez, G., Turgeon, D. (2019). *Research Activities on Plasma Technology at Canadian Nuclear Laboratories*. 24<sup>th</sup> Int. Symp. Plasma Chem. Naples, IT, June 9-14.

**Acknowledgments:** This study was funded by Atomic Energy of Canada Limited, under the auspices of the Federal Nuclear Science and Technology Program.



# Effect of crosslinker on the wettability and mechanical properties of hydrophobic coatings deposited via atmospheric pressure plasma

C. Rendon-Piedrahita<sup>1\*</sup>, K. Baba<sup>1</sup>, R. Quintana<sup>1</sup>, J. Bardon<sup>1</sup>, R. Heyberger<sup>2</sup>, J. Borek-Donten<sup>2</sup> and P. Choquet<sup>1</sup>

<sup>1</sup> Materials Research and Technology (MRT) Department, Luxembourg Institute of Science and Technology, Esch/Alzette, Luxembourg.

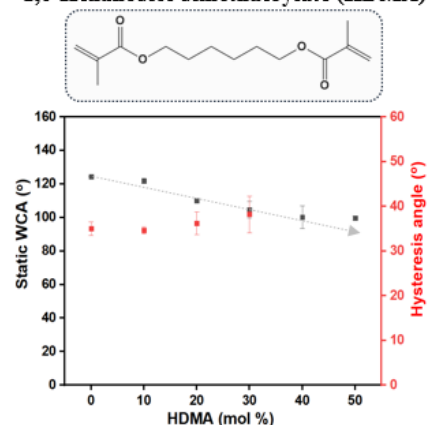
<sup>2</sup> Molecular Plasma Group (MPG), Foetz, Luxembourg

## Abstract:

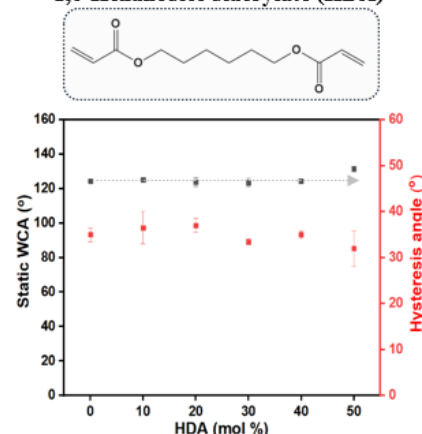
Hydrophobic coatings based on dodecyl acrylate (DOCA) and perfluorodecyl acrylate (PFDA) have been deposited via a single step, solvent-free and scalable atmospheric pressure plasma process. At certain composition, these coatings exhibit a static water contact angle (WCA) of around 125° accompanied by poor mechanical strength. In order to improve the coatings strength, two different chemical crosslinkers i.e., hexanediol methacrylate (HMDA) and hexanediol diacrylate (HDA), have been incorporated into the precursor mixtures. After plasma polymerization, both kind of coatings with crosslinkers admixture showed different wettability behaviour (Figure 1). The addition of HDMA reduces the WCA while increasing the mechanical strength, while the addition of HDA retains of the initial WCA magnitude while also increasing the mechanical strength. These changes are related to the variation of the surface free energy (SFE) and to the topography of the plasma coatings. The SFE was calculated with the Owens, Wendt, Rabel and Kaelble (OWRK) method and the roughness parameters were calculated at two different scales with two techniques: confocal microscopy and atomic force microscopy (AFM). The plasma polymers structure and properties have been studied via fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). Mechanical properties of the coatings has been quantified via nanomechanical analysis (AFM) and complemented with scratch testing (nano-scratch).

**Keywords:** atmospheric pressure plasma; cross-linkers, hydrophobicity, indirect discharge, mechanical strength.

### 1,6-Hexanediol dimethacrylate (HDMA)



### 1,6-Hexanediol diacrylate (HDA)



**Figure 1:** Illustration of the two different wettability behavior for each of the chemical crosslinkers.

# Solution Plasma for Enhanced Phosphorus Doping in TiO<sub>2</sub> Nanoparticles

C. Chokradjaoren<sup>1</sup>, J. Niu<sup>1</sup>, N. Saito<sup>1,2,3,4\*</sup>

<sup>1</sup>Department of Chemical Systems Engineering, Graduate School of Engineering, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

<sup>2</sup>Conjoint Research Laboratory in Nagoya University, Shinshu University, Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

<sup>3</sup>Japan Science and Technology Corporation (JST), Open Innovation Platform with Enterprises, Research Institute and Academia (OPERA), Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

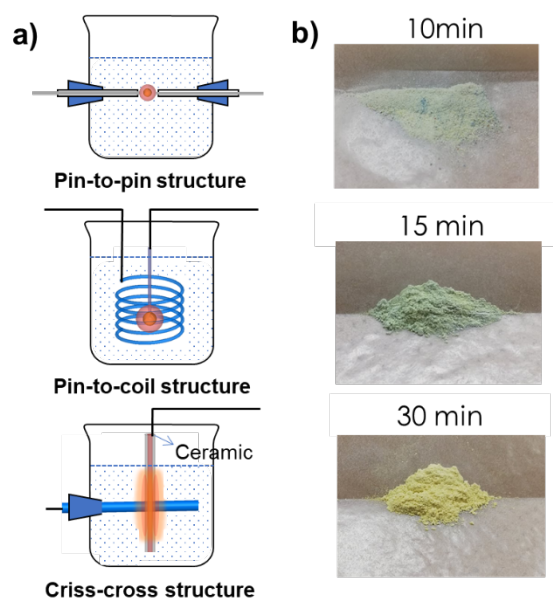
<sup>4</sup>Japan Science and Technology Corporation (JST), Strategic International Collaborative Research Program (SICORP), Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

## Abstract:

TiO<sub>2</sub> is considered as the best photocatalyst due to its attractive optical and electronic properties, low cost, non-toxicity, and long-term stability [1]. However, its band gap energy (3.0–3.2 eV) can only be excited in the UV light region, which accounts a small fraction (8%) of the solar energy, compared to the visible light (45%) [2]. It has been studied and reported that the desired band gap of TiO<sub>2</sub> nanoparticles can be tuned by using anionic dopant species rather than metals ions [1]. Doping of anionic species, such as nitrogen and phosphorus, were found to be most effective because its p states contribute the band gap narrowing [2]. Recently, several doping processes have proposed, however, they resulted in only small amounts ( $\leq 4\%$ ).

We have developed and investigated an alternative synthesis route that leads to increased phosphorus dopant concentration in TiO<sub>2</sub> nanoparticles, by electrical discharge in liquids, so-called solution plasma (SP). It was found that the SP could result in oxygen vacancy and incorporate the phosphorus, atoms into the structure of TiO<sub>2</sub>. Moreover, different configurations of discharges, pin-to-pin, pin-to-coil, and crisscross, as shown in **Figure 1**, were used to obtain different plasma mode, respectively. They provide different energy of particles that proceed the reaction in plasma field, which was found to relate to dopant concentration.

**Keywords:** solution plasma, TiO<sub>2</sub>, nanoparticles, phosphorus, doping, oxygen vacancy



**Figure 1:** Figure illustrating the solution plasma (SP) with (a) different configurations including pin-to-pin, pin-to-coil, and crisscross, and (b) the color change of phosphorus-doped TiO<sub>2</sub> prepared by the SP with pin-to-coil configuration at the different plasma treatment time (e.g., 10 min, 15 min, and 30 min).

## References:

1. Nyankson, E., Efavi, J., Agyei-Tuffour, B., Manu, G. (2021), *RSC Adv.*, 11, 17032.
2. Burda, C., Lou, Y., Chen, X., Samia, A. (2003), Enhanced Nitrogen Doping in TiO<sub>2</sub> Nanoparticles, *Nano Lett.*, 3, 8, 1049-1051.



## Adaptive Plasmas and Recent Developments in Plasma Medicine

Michael Keidar

The George Washington University, Washington DC 20052

E-mail: [keidar@gwu.edu](mailto:keidar@gwu.edu)

### Abstract

The uniqueness of plasma is in its ability to change composition *in situ*. Plasma self-organization could lead to formation of coherent plasma structures. These coherent structures tend to modulate plasma chemistry and composition, including reactive species, the electric field and charged particles. Formation of coherent plasma structures allows the plasma to adapt to external boundary conditions, such as different cells types and their contextual tissues. In this talk we will explore possibilities and opportunities that the adaptive plasma therapeutic system might offer. We shall define such an adaptive system as a plasma device that is able to adjust the plasma composition to obtain optimal desirable outcomes through its interaction with cells and tissues. Plasma adaptivity is based on the ability of measuring the cellular response to plasma action in real time and modifying the composition and power of plasma via a feedback mechanism. Plasma self-adaptation might be feasible due to self-organization and pattern formation when plasma interacts with targets. Recently, utilising a new treatment approach that eliminate reactive oxygen and nitrogen species, we observed the strong killing effect on melanoma and glioblastoma cells by the physical mechanisms. This modality of cell death is characterized by the leaking of bulk water from distinct points on the cellular membrane with the subsequent shrinkage of the cytoplasm. In this talk, we will describe these new observations and potential mechanisms.

# The plasma produced long-lived RONS as allies in biomedicine and food storage.

E. Sardella

<sup>1</sup> Istituto di Nanotecnologia, CNR-NANOTEC, c/o Dipartimento di Chimica, Università di Bari, Via Orabona 4, Bari, Italy

## Abstract:

Nowadays, cold atmospheric plasmas (CAP) are widely used in the field of biomedicine and food processing for their ability to promote, at room temperature, a selective effect against pathogens, without altering the functionality of healthy cells or food quality [1,2]. Currently, CAP have wide applications both when directly or indirectly applied [3]. The indirect approach consists of delivering exogenous reactive oxygen and nitrogen species (RONS) to cells/tissues or food through plasma treated liquids, hydrogels or gases [4]. In recent years it has been assessed that RONS produced by plasma are the main responsible of the observed effects. Often, by comparing the two approaches (i.e. direct vs indirect), due to the similarity of the observed results, it has been supposed with a good approximation that long-lived RONS more than short-lived ones are involved in the observed results [5] demonstrating that indirect approaches are valid alternatives to direct ones. In general, to assess the effective role of stable RONS against pathogens and malignant cells, are required: 1) valid methods for the analysis of RONS in complex media; 2) careful evaluations of the right correlation between observed phenomena and chemical composition of the liquid (gas) treated by plasma. In this research, current advances and outlooks on diagnostic analyses of RONS is proposed. The effects of Plasma Treated Water Solutions (PTWS) against cancer cells and of Plasma Treated Gases (PTG) against food pathogens contained in the artisanal daily products like ricotta cheese will be described.

In case of exposure of eukaryotic cells to PTWS it has been demonstrated that nitrite anions ( $\text{NO}_2^-$ ) instead, present in the PTWS, were found to improve the selective death of cancer cells (i.e. Saos2) compared to hybrid endothelial cells (i.e. EA.hy926) by decreasing the cytotoxic threshold of  $\text{H}_2\text{O}_2$  to non-toxic values for the endothelial cell line [6]. It is well established in fact that cancer cells can fuse with endothelial cells to form hybrid cells spontaneously, which facilitates cancer cells traversing the endothelial barrier to form metastase. It was also demonstrated that the pres-

ence of not negligible amount of aromatic biomolecules in PTWS could exacerbate the effects promoted by plasma produced RONS.

The observed results highlight an active role of long-lived reactive nitrogen species (i.e.  $\text{NO}$ ,  $\text{NO}_2^-$ ,  $\text{NO}_3^-$ ) respect to oxygen ones (i.e.  $\text{H}_2\text{O}_2$ ). On the other hand there is a growing body of literature that recognizes the importance of ROS for inactivation of microorganisms. However, very little attention has been paid to the role of RNS in bacterial deactivation. In our research it has been demonstrated that the use of PTG blownd on ricotta cheese is effective to control proliferation of pseudomonas spp., enterobacteriaceae and yeasts [7]. The PTG was obtained by mixing nitrogen with 10% of pure oxygen in order to maximize the content of RNS in the PTG. The sensory quality of the food was preserved after treatment, with a shelf-life prologation between 50% and 100% depending to the type of treatment and initial microbial contamination. These results show one of the first example of “gentle” and efficacious treatment of fresh perishable products by PTG without loss of nutrients and organoleptic quality of food.

**Keywords:** Cold Atmospheric Plasmas (CAP), plasma treated water solutions (PTWS), plasma treated gas (PTG), plasma medicine, plasma processing of food.

## References:

1. Adamovich, I.; Baalrud, S.D.; Bogaerts, A.; Bruggeman, P.J.; Cappelli, M.; Colombo, V.; Czarnetzki, U.; Ebert, U.; Eden, J.G.; Favia, P.; et al. (2017) The 2017 Plasma Roadmap: Low temperature plasma science and technology. *J. Phys. D. Appl. Phys.* 50, 323001.
2. Paula Bourke,1,Dana Ziuzina,1,@ Daniela Boehm,1,@ Patrick J. Cullen,2 and Kevin Keener3; Trends in Biotechnology, June 2018, Vol. 36, No. 6
3. Ratovitski, E.A.; Cheng, X.; Yan, D.; Sherman, J.H.; Canady, J.; Trink, B.; Keidar, M. (2014) Anti-cancer therapies of 21st century: Novel approach to treat human cancers using cold atmospheric

- plasma. *Plasma Process. Polym.* **11**, 1128–1137
4. Labay, C.; Roldán, M.; Tampieri, F.; Stancampiano, A.; Bocanegra, P.E.; Ginebra, M.P.; Canal, C. (2020) Enhanced generation of reactive species by cold plasma in gelatin solutions for selective cancer cell death. *ACS Appl. Mater. Interfaces*, **12**
  5. Veronico, V.; Favia, P.; Fracassi, F.; Gristina, R.; Sardella, E. (2021) The active role of organic molecules in the formation of long-lived reactive oxygen and nitrogen species in plasma-treated water solutions. *Plasma Process. Polym.*, e2100158
  6. Sardella, E.; Veronico, V.; Gristina, R.; Grossi, L.; Cosmai, S.; Striccoli, M.; Buttiglione, M.; Fracassi, F.; Favia, P. (2021) Plasma treated water solutions in cancer treatments: The contrasting role of rns. *Antioxidants* **10**
  7. E. F. Ricciardi, M. A. Del Nobile, A. Conte, F. Fracassi, E. Sardella; (2022) Effects of plasma treatments applied to fresh ricotta cheese; *Innovative Food Science & Emerging Technologies*, 102935.

## With Plasma from Medicine to Hydrogen

K.D. Weltmann

T.v. Woedtke, J.F. Kolb, R. Brandenburg, T. Gerling, S. Bekeschus, R. Bansemer, V. Hahn, J. Ehlbeck,

H. Brust, K. Zocher, V. Brüser, A. Kruth, R. Clemen

Leibniz Institute for Plasma Science and Technology, Greifswald Germany

Email: [weltmann@inp-greifswald.de](mailto:weltmann@inp-greifswald.de)

### Abstract

The lecture will focus on Cold Atmospheric Plasma Technology (CAP) as cross sectional technology and will give an overview of the multitude of possible applications beyond the known classic technical areas. CAP is a multifunctional tool which has made its way into ever-new areas of application and which can make a major contribution to solving society's challenges. Plasma technology offers direct and indirect solutions for current tasks in a wide variety of fields ranging from medicine, life science, agriculture, food production up to the generation of green energy.

The lecture will discuss the following areas:

- Tailor-made plasma sources as the basis for all applications
- Medicine: wound healing; oncology; tissue disinfection; implant surfaces
- Hygiene: plasma effectivity against microorganisms; treatment of air, water and surfaces
- Potential of CAP to face challenges in agriculture and food industry
- Disintegration of biomass for material and energetically exploitation
- CAP for green energy

The presentation is based on INP's research and development in an international context.

# Plasma-Induced Graft Polymerization of Polyethylenimine onto Polycaprolactone Composite Membrane for Heavy Metal Pollutants Treatment in Industrial Wastewater

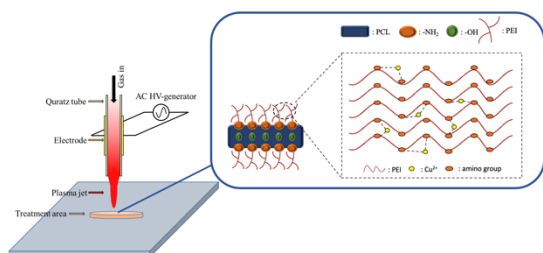
Sung-Lin Tu, Jason H.C. Yang\*

Department of Fiber and Composite Materials, Feng Chia University, Taichung, Taiwan

## Abstract:

As Taiwan taking the leading position in the global chip-manufacturing industry<sup>[1]</sup>, it also bears the burden of seeking treatments for heavy metal pollutants existing in wastewater. Conventionally, electroagulation<sup>[2]</sup>, chemical precipitation<sup>[3,4]</sup>, physical filtration<sup>[5]</sup>, and adsorption<sup>[6]</sup> are adopted with the possibilities of generating secondary pollutants. The present study manifests an innovative and green approach to graft metal ion adsorbent, polyethylenimine (PEI), onto electrospun polycaprolactone (PCL) composite membrane via atmospheric pressure nitrogen plasma grafting polymerization. FTIR absorption peak at  $\sim 1690\text{ cm}^{-1}$  is attributed to the amide group from PEI. Since the plasma exposure time is a dependent factor of  $\text{-NH}$  bond formation, an increased nitrogen content up to 3.3% is observed with extensive reaction time under plasma treatment. In addition, N1s spectra show a distinctive PEI dominating characteristic at 401.7eV, which suggest a successful grafting of PEI on the PCL membrane. According to the EDX analysis, significant amount of copper ions has been detected in PCL-PEI as compared to PCL membranes. This study has shown that a greener waste water treatment can be realized with the developed synthesis technology.

**Keywords:** atmospheric pressure plasma, electrospinning, polyethylenimine (PEI), adsorption.



**Figure 1:** Schematic of plasma-induced grafting of polyethylenimine onto polycaprolactone membrane demonstrates metal-ion adsorption capability.

## References:

1. Crawford, Alan, et al. "The World Is Dangerously Dependent on Taiwan for Semiconductors." Politics, Bloomberg, 26 January, 2021, <https://www.bloomberg.com/news/features/2021-01-25/the-world-is-dangerously-dependent-on-taiwan-for-semiconductors>.
2. Al-Shannag, M., et al. (2015). Heavy metal ions removal from metal plating wastewater using electrocoagulation: Kinetic study and process performance. *Chemical Engineering Journal*, 260, 749-756.
3. Fedje, K. K., et al. (2019). Enhanced soil washing with copper recovery using chemical precipitation. *Journal of Environmental Management*, 236, 68-74.
4. Yan, J., et al. (2019). An integrated process of chemical precipitation and sulfate reduction for treatment of flue gas desulphurization wastewater from coal-fired power plant. *Journal of Cleaner Production*, 228, 63-72.
5. Ghaemi, N., et al. (2015). Polyethersulfone membrane enhanced with iron oxide nanoparticles for copper removal from water: application of new functionalized  $\text{Fe}_3\text{O}_4$  nanoparticles. *Chemical Engineering Journal*, 263, 101-112.
6. Igberase, E., et al. (2014). The adsorption of copper (II) ions by polyaniline graft chitosan beads from aqueous solution: equilibrium, kinetic and desorption studies. *Journal of Environmental Chemical Engineering*, 2(1), 362-369.

## Application of continuous flow plasma brushes for inactivation of antibiotics from communal and industrial wastewaters

W. Babinska<sup>1</sup>, A. Motyka-Pomagruk<sup>1</sup>, D. Terefinko<sup>2</sup>, M. Caban<sup>3</sup>, P. Jamroz<sup>2</sup>, P. Pohl<sup>2</sup>, E. Lojkowska<sup>1</sup>, W. Sledz<sup>2</sup>, A. Dzimitrowicz<sup>2</sup>

<sup>1</sup>Intercollegiate Faculty of Biotechnology University of Gdansk and Medical University of Gdansk, University of Gdansk, Gdansk, Poland

<sup>2</sup>Faculty of Chemistry, Wrocław University of Science and Technology, Wrocław, Poland

<sup>3</sup>Faculty of Chemistry, University of Gdansk, Gdansk, Poland

### Abstract:

Currently, it is important that urban and industrial wastewaters contribute as huge reservoirs of antibiotic resistance to the persistence of difficult to combat bacterial pathogens. In the timeframe of 2014 and 2016, approximately one million people died due to infections caused by antibiotic resistant bacteria through inefficient medication, or lack of any available treatment (Alexander et al., 2020). Thus, implementation of advanced wastewater cleaning processes that are capable of decomposing biologically active drugs, into the conventionally applied wastewater treatments is an important step to protect the aquatic environment from dispersal of multidrug resistance resistance determinants among free-living and pathogenic microbes.

A remedy to these problems might be application of cold atmospheric pressure plasma (CAPP)-based technology. To reach this aim, we have constructed two, continuous flow, plasma brushes, dedicated to degrade antibiotics such as: ciprofloxacin, ofloxacin, enrofloxacin, doxycycline, chloramphenicol, and trimetoprim, as well as the mixture of these antibiotics. In the developed by us plasma brushes, either pulse-modulated radio-frequency atmospheric pressure glow discharge (pm-rf-APGD) or dielectric barrier discharge (DBD) was applied as a plasma source, allowing to ignite the CAPP in the form of several plasma cones.

To check whether these devices might be used in the wastewater purification process, the several analyses such as determination of total organic carbon (TOC), total nitrogen (TN) concentration in addition to High-Performance Liquid Chromatography-Diode Array Detection (HPLC-DAD), were used for determining the antibiotics removal from liquid disposals. Additionally, to investigate whether plasma brush might be applied for limitation of antibiotic resistance in the environment, the disc-diffusion assays of comparing antibacterial properties of untreated and pm-rf-APGD- or DBD-treated antibiotics solutions towards 7 bacterial strains were performed.

The conducted research showed that the treatment of the selected antibiotics solutions with cold atmospheric plasma of the pm-rf-APGD- or DBD-type changed the values of TOC and TN, which indicated decomposition of a stated antibiotic. Next, by using HPLC-DAD, we confirmed that application of pm-rf-APGD plasma brush led to a 91.4% efficiency of removal for trimetoprim up to 100% for doxycycline. Quite lower efficiencies of removal were observed where DBD plasma brush was used for antibiotics purification from liquid disposals. In this case, the removal efficiency was found to be 49.8% for chloramphenicol and 65.7% for doxycycline, but analyzed from the solution, being a mixture of antibiotics. Additionally, it was found that the antibacterial properties of the aqueous solutions of antibiotics from fluoroquinolones group treated by pm-rf-APGD plasma brush, dropped achieving up to 100% reduction for the solution of ofloxacin or ciprofloxacin towards *Staphylococcus hemolyticus* and minimum 36.3% of ciprofloxacin towards *Salmonella typhimurium*. In the case of application of DBD plasma brush the maximum reduction of 100% against *Serratia marcescens* and minimum 38.2% towards *Escherichia coli* for mixture of 4 antibiotics were determined.

In the view of application of CAPP-based continuous flow plasma brushes in the wastewater purification, we truly believe that this method will be competitive to advanced oxidation process (AOP) including Fenton's reactions.

**Keywords:** antibiotic resistance, human pathogenic bacteria, plasma brush, cold atmospheric pressure plasma

### References:

1. Alexander, J., Hembach, N. & Schwartz, T. (2020), Evaluation of antibiotic resistance dissemination by wastewater treatment plant effluents with different catchment areas in Germany, *Sci Rep*, 10, 8952.



*Acknowledgement:* This work was supported by National Science Centre, Poland, *via* Sonata 15 project (UMO-2019/35/D/ST8/04107).

# The Physical Effects of Plasma Medicine on Cells: Radio Frequency Stimulated Intercellular and Intracellular Mechanical Waves

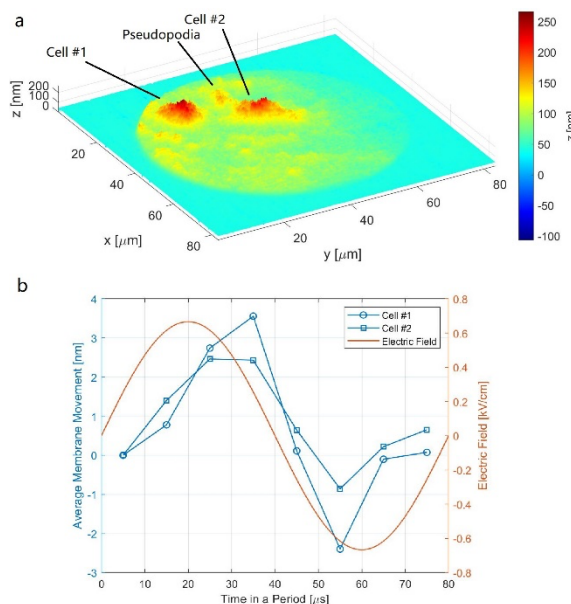
L. Lin<sup>1</sup>, X. Yao<sup>1</sup>, D. Yan<sup>1</sup>, M. Keidar<sup>1</sup>

<sup>1</sup> Department of Mechanical and Aerospace Engineering, School of Engineering and Applied Science, The George Washington University, Washington DC, USA

## Abstract:

Plasma medicine, such as cancer therapies and wound treatments, is currently one of the top applications of cold atmospheric plasmas (CAP). In the last decade of research works, reactive oxygen and nitrogen species (RONS) are believed to be the major role of these applications. This thus raises the indirect treatments that use the plasma-treated solutions or drugs rather than treat the targeting cells or tissues directly with CAP. However, the difference between direct and indirect treatments cannot be ignored. Two explanations have been reported: 1) the CAP treated solution may not include some short lifetime species; 2) the indirect treatment does not include the electromagnetic (EM) radiation from CAP. The latter is thus named as the physical effects of plasma medicine. Due to the surface charge on the cell membrane, the membrane oscillation under the external electric field has been reported. In this work, we developed a Michelson laser interferometer system coupled with a high-speed ICCD camera and a radio frequency (RF) emission system to capture the cell membrane and the propagation of the resulting mechanical waves. The system can observe such an oscillation in 3D and real-time. As shown in Figure 1a, two cells are captured as an example, and by averaging the temporally resolved height over the cell region, we observed the cell thickness oscillations are in-phase with the external RF electric field. In the further analysis, we will discuss the propagation of such an oscillation, mainly a transverse wave along the cell membrane from the center to its pseudopodia, and the wave in the extracellular medium. The corresponding cancer cell viability and sensitization to cancer medicines are also analyzed. The physical effects of species transportations on the cell membrane are thus suggested.

**Keywords:** plasma medicine, cell membrane, cell imaging, ion channel, apoptosis, sensitization.



**Figure 1:** A real-time 3D cell imaging. a, Two cells are shown as an example. b, The average cell height variation over time, which are in-phase with the external electric field, known as the vertical membrane oscillation.

## References:

1. Laroussi, M. (2018) Plasma medicine: A brief introduction, *Plasma*, 1, 47-60.
2. Firdman, G., Friedman, G., Gutsol, A., Shekhter, A. B., Vasilets, V. N., Fridman, A. (2008) Applied plasma medicine, *Plasma Process. Poly.*, 5, 503-533.
3. Yao, X., Lin, L., Soni, V., Gjika, E., Sherman, J. H., Yan, D., Keidar, M. (2020) Sensitization of glioblastoma cells to temozolomide by a helium gas discharge tube, *Phys. Plasmas*, 27, 114502.
4. Yan, D., Wang, Q., Adhikari, M., Malyavko, A., Lin, L., Zolotukhin, D. B., Yao, X., Kirschner, M., Sherman, J. H., Keidar, M. (2020) A physically triggered cell death via transbarrier cold atmospheric plasma cancer treatment, *ACS Appl. Mater. Interfaces*, 12, 34548-34563.

## Antibacterial, biodegradable nanohybrids for therapy of chronic wounds.

A. Manakhov<sup>1\*</sup>, E. Permyakova<sup>1</sup>, A. Solovieva<sup>1</sup>

<sup>1</sup> Research Institute of Clinical and Experimental Lymphology – Branch of the ICG SB RAS, 2 Timakova st., 630060 Novosibirsk, Russian Federation

### Abstract:

The therapy of chronic wounds is difficult since this pathology is characterized by multifactorial homeostatic disorders at the systemic and local levels. Therefore, therapeutic approaches to the treatment of diabetic foot syndrome should combine several factors: support and stimulation of the synthesis of natural extracellular matrix and its protection from destruction by metalloproteinases, stimulation of angiogenesis and migration of specialized cell types to the damaged area. Combining these factors can be achieved by using polymeric biocompatible matrices as wound dressings, which can be functionalized by biomolecules and promote cell adhesion, stimulating their migration, proliferation, and differentiation. Plasma, enriched with platelet lysate (PRP), is a natural autologous balanced ensemble of growth factors, extracellular matrix components, and proteoglycans. PRP promotes proliferation, migration of stem cells, attracting macrophages to the damaged area, regulates the cytokine background of the wound, limiting inflammation and promoting the growth of new capillaries [1]. It is actively used in various medical fields, including treating chronic non-healing ulcers in type 2 diabetes. However, the results of the use of PRP for the treatment of chronic wounds show different efficacy. Perhaps the low efficiency of this therapy in some patients is associated with an imbalance in the natural ensemble of active PRP factors. In addition, it is necessary to maintain optimal moisture and wound sanitation since most of them are characterized by hyperexudation and the presence of bacterial biofilms. Despite the long-term active search for highly effective materials for this purpose, the problem has not yet been solved. This work is devoted to developing and studying modified polycaprolactone nanofibers using

plasma polymerization to immobilize the compositional composition of PRP therapy for complex healing wounds in type 2 diabetes mellitus with the additional use of a superabsorbent layer to optimize wound moisture.

It is known that toxic compounds, malonic anhydride, peroxyxynitrites, and oxidized lipids accumulate in the blood in type 2 diabetes. On the other hand, the natural cocktail contains numerous cytokines, extracellular matrix proteins, and growth factors necessary for successful regeneration. The listed compounds are of a protein nature, i.e. contain amide groups that can be attached to the COOH groups of the nanofiber surface. In turn, toxic compounds are aldehydes, and nitrites do not bind to them.

Our results showed that the combination of antibacterial superabsorbent polymer with a plasma modified PCL nanofibers with grafted PRP allows to speed up the closure of the wounds significantly, as the *in vivo* results have revealed. The developed material has vast potentials for biomedical applications.

**Acknowledgments:** Authors gratefully acknowledge the financial support of the Russian Science Foundation (grant No. 18-75-10057)

**Keywords:** Nanofibers, XPS, wound healing.

### References:

1. Solovieva, A.; Miroshnichenko, S.; Kovalskii, A.; Permyakova, E.; Popov, Z.; Dvořáková, E.; Kiryukhantsev-Korneev, P.; Obrosova, A.; Polčák, J.; Zajíčková, L.; et al. Immobilization of Platelet-Rich Plasma onto COOH Plasma-Coated PCL Nanofibers Boost Viability and Proliferation of Human Mesenchymal Stem Cells. *Polymers*, **2017**, 9, 736.

## **Tribology 2022 Virtual session: Biotribology/ Green Tribology**

# Soft tribology, biotribology and viscoelastic lubrication: interpretation and application

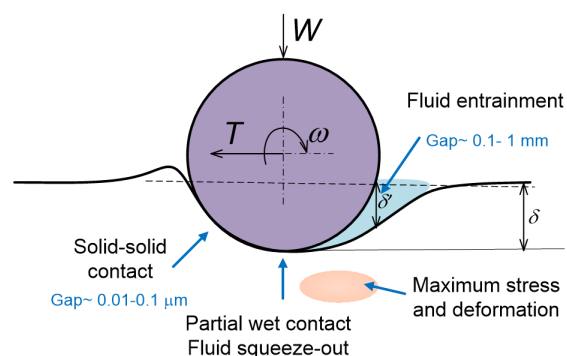
J.R. Stokes, Y. Xu

School of Chemical Engineering, The University of Queensland, Brisbane, Australia

## Abstract:

Soft contact tribology is emerging as a useful tool in biotribology for the design of foods and beverages due to its relevance to oral processing and oral sensory perception<sup>1</sup>, as well as for biomedical applications<sup>2</sup>. Soft tribology differs from traditional studies in elastohydrodynamic lubrication due to the highly deformable and viscoelastic nature of the surfaces involved. We highlight our recent finding that the combination of normal and lateral forces results in an asymmetric deformation that is not considered in the Hertzian contact model that is traditionally used to predict normal deformation and to construct the scaling parameter in Stribeck-Hersey analysis<sup>3</sup>. We believe this asymmetric deformation promotes entrainment of lubricant into the contact under low-speed conditions where lubricant is traditionally considered to be excluded from the contact. We discuss how this new model explains many anomalous results in soft-tribology and how it is assisting progress towards development of a universal scaling model akin to the Stribeck curve. In addition, we aim to highlight how our fundamental investigations into viscoelastic lubrication improve interpretation of measured tribological responses of gels that are commonly studied for biomedical applications<sup>4</sup>, and soft lubrication studies involving many complex and multiphase fluids including emulsions and particle suspensions<sup>5</sup>. We also highlight progress in utilisation of soft tribology in predicting sensory perception of foods and beverages<sup>1</sup>.

**Keywords:** soft tribology, oral tribology, contact mechanics, rheology, soft matter, colloids, oral processing, sensory.



**Figure 1:** A schematic on the load and deformation effect of soft contact lubrication in the boundary-mixed regime.

## References:

1. HM Shewan, C Pradal, JR Stokes (2020) Tribology and its growing use toward the study of food oral processing and sensory perception, *Journal of Texture Studies* 51 (1), 7-22
2. C Pradal, GE Yakubov, MAK Williams, MA McGuckin, JR Stokes, Lubrication by biomacromolecules: mechanisms and biomimetic strategies (2019), *Bioinspiration & biomimetics* 14 (5), 051001
3. Y Xu, B Cartwright, L Advincula, C Myant, JR Stokes (2021) Generalised scaling law for soft contact tribology: Influence of load and asymmetric surface deformation, *Tribology International* 163, 107192
4. L Geurds, Y Xu, JR Stokes, Friction of lubricated hydrogels: Influence of load, speed and lubricant viscosity (2021) *Biotribology* 25, 100162
5. Y Xu, JR Stokes (2020) Soft lubrication of model shear-thinning fluids, *Tribology International* 152, 106541.

# Posters



# Modeling of surface morphology kinetics and nanostructures formation during binary thin film deposition

A. Galdikas, G. Kairaitis

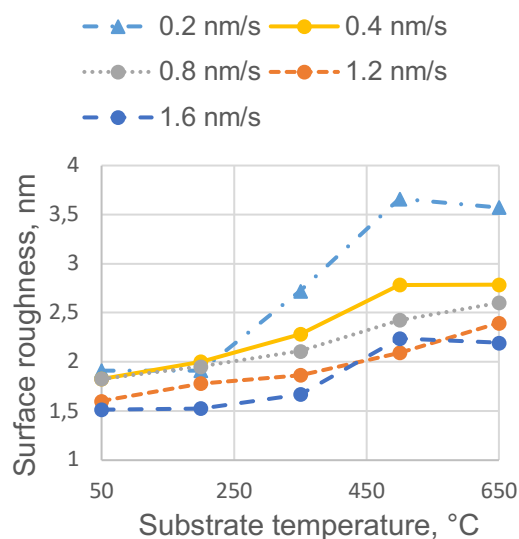
Physics Department, Kaunas University of Technology, Kaunas, Lithuania

## Abstract:

Influences of various kinetic factors such as substrate temperature, incoming ion flux on binary thin film phase structure, roughness and growth mechanisms were investigated by using a proposed mathematical model. The mathematical model includes the processes of adsorption, phase separation and diffusion due to surface curvature of a growing film. The process of phase separation is mathematically described by using the Cahn-Hilliard equation. The series of numerical experiments were performed to determine the impacts of substrate temperature, incoming ion flux to phase structure and surface roughness of binary thin films. To express the influence of substrate temperature on diffusion processes, temperature dependent diffusion coefficients, which are present in the mathematical descriptions of phase separation process and diffusion due to surface curvature of a growing film, were used in calculations. To include the impact of substrate temperature on adsorption process in the model, the dependencies of sticking coefficient on substrate temperature were adopted. The effect of incoming ion flux on diffusion processes was also included in the model. Observed relationships between surface roughness and substrate temperature at different growth rates are given in Figure 1. Relationships between average grain size and substrate temperature were noticed and discussed. The modeling results demonstrated that the increase in the substrate temperature resulted in the mostly monotonic increase in the surface roughness of thin films. The similar mostly monotonic relationships were reported between the size of nanoparticles formed and the substrate temperature. Moreover, the linear and nonlinear relationships (depending on growth rate) between surface roughness and average size of nanoparticles were also discovered, so the increase in the surface roughness caused by an increase in substrate temperature was considered to be influenced by the increase of nanoparticle size. The formation of columns and layers can occur if other processes such as diffusion and segregation take place. In this paper the main attention is paid on the formation of multilayered

structures during binary components co-deposition, which is experimentally observed but the mechanism of formation is not well understood. Very important processes are surface segregation and the depth dependent diffusion taking place during binary films deposition process which are responsible for various nanostructures formation.

**Keywords:** aluminum co-doped scandium stabilized zirconia; phase separation; kinetic modeling; thin films; surface roughness; compounds.



**Figure 1:** Plots of surface roughness versus substrate temperature obtained by using different growth rates.

## References:

1. Kairaitis, G.; Galdikas, A. (2020), Modelling of phase structure and surface morphology evolution during compound thin film deposition, *Coatings*, 10, 1077. <https://doi.org/10.3390/coatings10111077>.
2. Galdikas, A.; Sriubas, M.; Kairaitis, G.; Virbukas, D.; Bockute, K.; Galdikas, M.; Moskalioviene, T.; Laukaitis, G. (2022), Investigation of Morphology of Aluminum Co-Doped Scandium Stabilized Zirconia (ScAlSZ) Thin Films, *Coatings*, 12, 31. <https://doi.org/10.3390/coatings12010031>

## Development of functional coatings with anti-abrasion and anti-icing properties

J. P. Sousa, Y. Kolen'ko

International Iberian Nanotechnology Laboratory, Braga, Portugal

### Abstract:

Polymer coatings have been explored as possibilities for protective applications, in order to increase the abrasion resistance of different substrates. There are two main ways to improve the abrasion resistance of coatings: one is to optimize the polymer lacquer components, and the other is to reinforce the coatings by embedding fillers into them. Nanomaterials represent a promising groups of fillers, these materials have been shown to improve the mechanical properties even at low loadings and, due to their small particle size, they do not affect the transparency of clear-coats. Different hard particles have been used in formulations to impart greater abrasion strength. These included zirconia particles, which can improve scratch and abrasion resistance of the obtained coatings. Zirconia materials ( $\text{ZrO}_2$ ) has numerous unique properties, such as good mechanical strength, excellent resistance against crack propagation, and good thermal resistance. Based on these properties, in this work  $\text{ZrO}_2$  materials were incorporated in a polymeric solution in order to improve the mechanical properties such as hardness and abrasion resistance of glass substrates. The obtained coating present high transmittance values in infrared region. This work allowed the fabrication of antireflective coatings through the sol-gel route.

Generally, a surface can absorb water and wet snow which is able to condense into ice below  $0^\circ\text{C}$ , and then icing occurs on the surfaces. If water and wet snow can be repelled immediately, instead of sticking on the surface, the formation of frost or ice and accumulation of snow or slush are able to be avoided. Micro- and nanoscale hierarchical structures have proven to be vital in generating the superhydrophobic property of a coating surface. In this way, different nanostructured surfaces of metal oxides such as zinc oxide ( $\text{ZnO}$ ), titania, silica and copper oxide have been extensively studied. These nanoparticles create a nano scaled rough surface, which reduce the contact area between water and the solid surface. Among these materials,  $\text{ZnO}$  may be considered as an outstanding candidate of superhydrophobic coating because of its excellent mechanical durability, thermal stability, environmentally friendly perspectives

and low cost. A combination of both low surface energy and hierarchical rough structure need to achieve the  $\text{ZnO}$  superhydrophobic surfaces. The hierarchical structure of micro/nanoscale roughness of the needle-like  $\text{ZnO}$  nanomaterials decreases the wetted surface fraction and the triple contact line at the solid-liquid interface. Under this condition, water droplets are suspended by the air pockets trapped in the pits/cavities between needle-like  $\text{ZnO}$  and cannot penetrate the substrate surface. Thus, in this work it was fabricated a superhydrophobic micro/nano hierarchical zinc oxide surface on glass substrate. The polymeric solutions chosen to be use in this work were acrylates and polyurethanes. These polymeric solutions have excellent properties such as, excellent optical quality, impact resistance, crack propagation resistance, and solvent resistance.

**Keywords:** anti-abrasion, antireflective coatings, anti-icing, zirconia, micro/nano hierarchical structures

# Functional nanomaterial-based coatings for automotive industry

C. Ponte, J. P. Sousa

International Iberian Nanotechnology Laboratory, Braga, Portugal

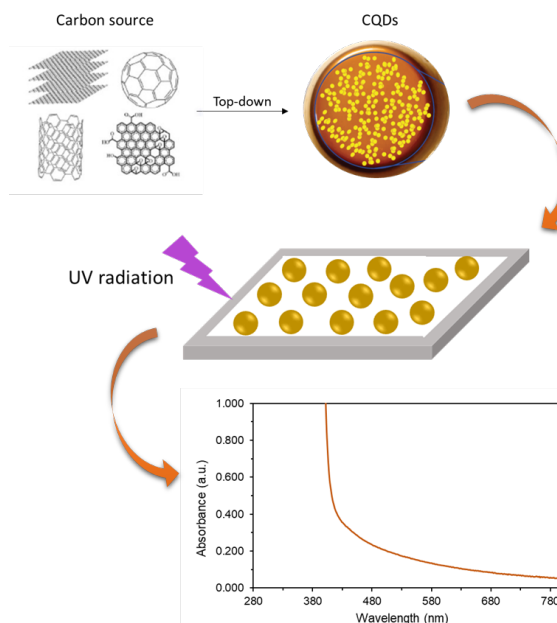
## Abstract:

One of the greatest challenges that automotive industry have been facing is the decrease of durability of car paints, induced by their extended exposure to ultraviolet (UV) radiation.<sup>1</sup> Nowadays, car paints already present UV protective compounds in their formulations, mainly inorganic UV-absorbing systems, such as titanium dioxide (TiO<sub>2</sub>), zinc oxide (ZnO) and cerium oxide (CeO<sub>2</sub>).<sup>2</sup> However, these materials struggle to address the current needs of this industry, due to their short UV absorption range and poor photostability, resulting on the deterioration of the polymeric matrices, and consequently leading to paint's discoloration, loss of adhesion and flexibility.<sup>3</sup> Henceforth, the development of novel and photostable UV-absorbers is of utmost importance towards a more efficient and sustainable painting process.

Over the past years, quantum dots (QDs) based-materials have been raising great interest, owing to their distinct features, such as excellent UV absorption and high transparency in the visible light range. Amongst several QDs reported, carbon QDs are currently gathering tremendous attention in reaserch due to their low toxicity, environmental sustainability, low-cost and simple synthetic routes, ease bandgap tunability through surface functionalization and/or metal doping, and high compatibility with different polymeric matrices.<sup>4</sup>

Keeping this in mind, within the scope of this work we prepared carbon QDs and modulate their optical properties with different functional groups and/or doping with distinct metal elements. Thereafter, we incorporated the carbon QDs with the most promosing optical properties in commercially available automotive paints as well as in new polymeric formulations. Finally, we evaluated the capacity of each prepared coating to absorb UV radiation by UV-Vis-NIR sprectoscopy and their resistance to UV irradiation under prolonged exposure.

**Keywords:** Automotive industry, Carbon quantum dots, UV absorption, automotive coatings.



**Figure 1:** Representative scheme of the three main goals of this work.

## References:

1. Zayat, M., Garcia-Parejo, P., Levy, D. (2007), Preventing UV-light damage of light sensitive materials using a highly protective UV-absorbing coating, *Chemical Society Review*, 36, 1270-1281.
2. Uthirakumar, P., Devendiran, M., Yun, J.-H., Kim, G. C., Kalaarasan, S. (2018), Role of carbon quantum dots and film thickness on enhanced UV shielding capability of flexible polymer film containing carbon quantum dots/N-doped ZnO nanoparticles, *Optical Materials*, 84, 771-777.
3. Liu, H., Hu, D., Chen, X., Ma, W. (2021), Surface engineering of nanoparticles for highly efficient UV-shielding composites, *Polymers for Advanced Technologies*, 32, 6-16.
4. Rasal, A. S., Yadav, S., Yadav, A., Kashale, A. A., Manjunatha, S. T., Altaee, A., Chang, J. Y. (2021), Carbon Quantum Dots for Energy Applications: A Review, *ACS Applied Nano Materials*, 4, 6515-6541.

# Synthesis and characterization of GaN/ReS<sub>2</sub>, ZnS/ReS<sub>2</sub> and ZnO/ReS<sub>2</sub> core/shell nanowire heterostructures

Edgars Butanovs<sup>1</sup>, Alexei Kuzmin<sup>1</sup>, Sergei Piskunov<sup>1</sup>, Krisjanis Smits<sup>1</sup>, Aleksandr Kalinko<sup>2</sup>, Boris Polyakov<sup>1\*</sup>

<sup>1</sup> Institute of Solid State Physics, University of Latvia, Kengaraga Street 8, Riga LV-1063, Latvia

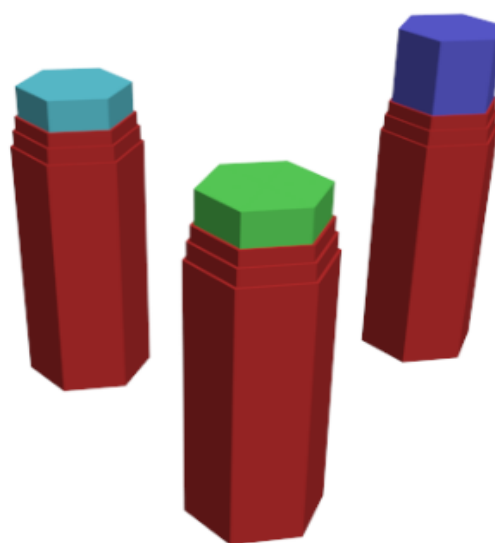
<sup>2</sup> Department Chemie, Naturwissenschaftliche Fakultät, Universität Paderborn, Warburger Strasse 100, 33098 Paderborn, Germany

## Abstract:

Layered 2D van der Waals (vdW) materials such as graphene and transition metal dichalcogenides have recently gained a great deal of scientific attention due to their unique properties and prospective applications in various fields such as electronics and optoelectronics, sensors and energy. As a direct bandgap semiconductor in both bulk and monolayer forms, ReS<sub>2</sub> stands out for its unique distorted octahedral structure that results in distinctive anisotropic physical properties; however, only a few scalable synthesis methods for few-layer ReS<sub>2</sub> have been proposed thus far. Here, the growth of high-quality few-layer ReS<sub>2</sub> is demonstrated via sulfurization of a predeposited rhenium oxide coating on different semiconductor material nanowires (GaN, ZnS, ZnO). As-produced core-shell heterostructures were characterized by X-ray diffraction, scanning and transmission electron microscopy, micro-Raman spectroscopy and X-ray absorption spectroscopy. Experimental characterizations were supported by total energy calculations of the electronic structure of ReS<sub>2</sub> nanosheets and GaN, ZnS, and ZnO substrates. Our results demonstrate the potential of using nanowires as a template for the growth of layered vdW materials to create novel core-shell heterostructures for energy applications involving photocatalytic and electrocatalytic hydrogen evolution.

**Keywords:** composite nanomaterials, heterostructures, core-shell nanowires, layered 2D materials, transition metal dichalcogenides, X-ray diffraction, electron microscopy, micro-Raman and X-ray absorption spectroscopy.

## Core-shell nanowires



**Figure 1:** Figure illustrating schematic representation of the core-shell GaN/ReS<sub>2</sub>, ZnS/ReS<sub>2</sub> and ZnO/ReS<sub>2</sub> core/shell nanowire heterostructures.

## References:

1. Butanovs, E., Kuzmin, A., Piskunov, S., Smits, K., Kalinko, A., Polyakov, B. (2021) Synthesis and characterization of GaN/ReS<sub>2</sub>, ZnS/ReS<sub>2</sub> and ZnO/ReS<sub>2</sub> core/shell nanowire heterostructures, *Applied Surface Science*, 536, 147841.

# Surface chemical and physical modification of PAM film induced by ion-beam irradiation for liquid crystal alignment

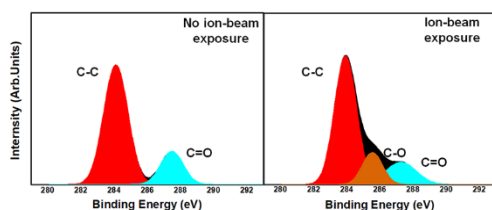
D. W. Lee<sup>1</sup>, J. H. Won<sup>1</sup>, D. H. Kim<sup>1</sup>, J. Y. Oh<sup>1</sup>, D.-S. Seo<sup>1,\*</sup>

<sup>1</sup> IT Nano Electronic Device Laboratory, Department of Electrical and Electronic Engineering, Yonsei University, Seoul, Republic of Korea

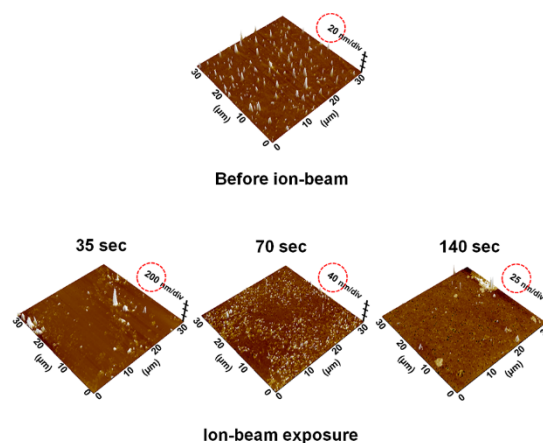
## Abstract:

The characteristics of ion beam (IB) irradiated polyarylamide (PAM) films were analyzed to determine their potential use as a liquid crystal (LC) alignment layer. The transmittance of the PAM films at various IB irradiation conditions was measured to verify the possibility of using it as an LC system. Anti-parallel LC cells with the IB-treated PAM films were fabricated to examine the LC alignment characteristics. Polarized microscopy and pre-tilt angle analyses confirmed uniform and homogeneous LC alignment. X-ray photoelectron spectroscopy (XPS) and atomic force microscopy (AFM) analyses were conducted to determine the effect of the IB irradiation on the film. After exposure to an IB irradiation, remarkable chemical composition modification of the PAM film, including the reduction of C=O bonds and the increasing of C-O bonds that affected the uniform LC alignment, was observed via the XPS analysis as presented in Figure 1. Furthermore, the AFM analysis revealed that low roughness value was obtained after IB irradiation as presented in Figure 2. Line profiles of PAM films before and after IB irradiation was also investigated in Figure 3. Twisted-nematic LC cell based on IB-treated PAM films show enhanced electro-optical performances. Therefore, we are convinced that the polymer films treated by IB irradiation show potential as alternative alignment layer in LC applications.

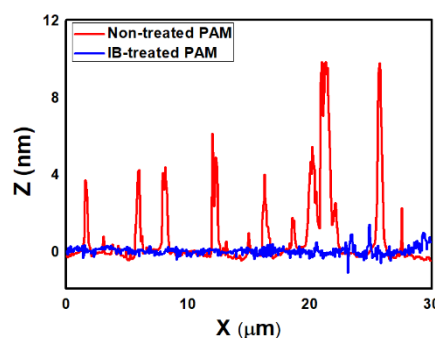
**Keywords:** polyarylamide, ion-beam irradiation, liquid crystal alignment, pre-tilt angle, electro-optical characteristic, atomic force microscopy, x-ray photoelectron spectroscopy.



**Figure 1:** X-ray photoelectron spectroscopy results of the polyacrylamide films before and after ion-beam irradiation.



**Figure 2:** Atomic force microscopy images of the polyacrylamide films according to ion-beam irradiation time (35, 70, and 140 sec).



**Figure 3:** Line profiles of polyacrylamide films before and after ion-beam treatment.

## References:

1. Chaudhari, P., Lacey, J.A., Lien, S.-C.A., Speidell, J.L. (1998) Atomic beam alignment of liquid crystals, *Jpn. J. Appl. Phys.*, 37, L55–L56.
2. Lee, J.J., Park, H.G., Han, J.J., Kim, D.H., Seo, D.S. (2013) Surface reformation on solution-derived zinc oxide films for liquid crystal systems via ion-beam irradiation, *J. Mater. Chem. C*, 1, 6824–6828.



# Self-improving of Protective Thin Films Based on Perhydropolysilazane

Elizaveta Shmagina \*, Matti Danilson, Valdek Mikli, Sergei Bereznev

Department of Materials and Environmental Technology, Tallinn University of Technology, Tallinn, Estonia

## Abstract:

Silicon dioxide thin films are a promising alternative to protective glass in solar cells. In this work, we investigate the effect of different curing conditions on the degree of perhydropolysilazane (PHPS) film conversion to  $\text{SiO}_2$ , which can be used as a matrix for a nanocomposite thin film protective coating. For that one-layer and four-layer thin films were spin-coated from 20% PHPS solution in dibutyl ether (durXtreme GmbH) onto the soda lime glass / Mo substrates, dried onto hotplate and cured by exposure to moderate temperatures ( $180^\circ\text{C}$ , 60 min) or UV light (40 min, 185nm+254nm wavelengths). Four-layer structures were obtained by cyclic repeating of the spin-coating / drying / curing stages.

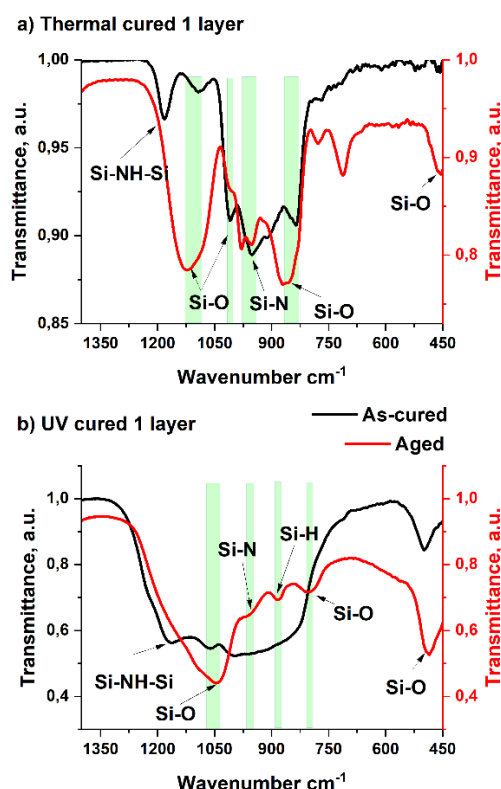
A number of research groups reported that thermal curing of PHPS produces a film that is completely converted to  $\text{SiO}_2$ , while UV curing gives only partial conversion [1]. However, UV curing is currently looks like the most promising method for converting PHPS due to its shorter duration and insignificant heating during curing compared to the thermal method [2].

The results of X-ray photoelectron spectroscopy and high resolution-scanning electron microscopy with energy dispersive X-ray analysis studies of our samples showed the formation of the films with composition close to (i)  $\text{SiO}$  for thermal curing method and (ii) films close to  $\text{SiO}_2$  for UV curing technique. However, the conversion of the UV cured film is not uniform in cross-section, which is especially noticeable for four-layer samples i.e. the process of complete conversion of obtained layers to  $\text{SiO}_2$  is not finished.

To study the hypothesis of non-complete curing, the ATR-FTIR spectroscopy was used after layers curing, as well as after cured layers aging in desiccator at room temperature for two months (Fig.1). The increase of the intensity of the peaks corresponding to  $\text{Si-O}$  bonds in comparison with other bonds in the spectra for aged samples vs. as-cured samples shows practically complete films conversion to  $\text{SiO}_2$ . Thus, it was found that additional “aging” of prepared structures improves significantly compositional uniformity of all obtained layers to  $\text{SiO}_2$ . This implies the need

for further optimization of the curing processes of PHPS thin films to achieve high uniformity of the resulting  $\text{SiO}_2$ .

**Keywords:** polysilazane, PHPS, thin films, thermal curing, UV curing, protective coating



**Figure 1:** ATR-FTIR spectra of the (a) thermal cured film and (b) UV cured film.

## References:

1. Morlier, A., Cros, S., Garandet, J.P., Alberola, N. (2014) Structural properties of ultra-violet cured polysilazane gas barrier layers on polymer substrates, *Thin Solid Films.*, 550, 85–89.
2. Naganuma, Y., Horiuchi, T., Kato, C., Tanaka, S. (2013) Low-temperature synthesis of silica coating on a poly(ethylene terephthalate) film from perhydropolysilazane using vacuum ultraviolet light irradiation, *Surface and Coatings Technology.*, 225, 40–46.



# Fabrication Multilayer Nanocoating for Marine Applications

M. Caruso<sup>1</sup>, A. Corozzi<sup>1</sup>, F. Veronesi<sup>1</sup>, M. Raimondo<sup>1</sup>, V. Piazza<sup>2</sup>, F. Garaventa<sup>2</sup>, F. Castelli<sup>2</sup>

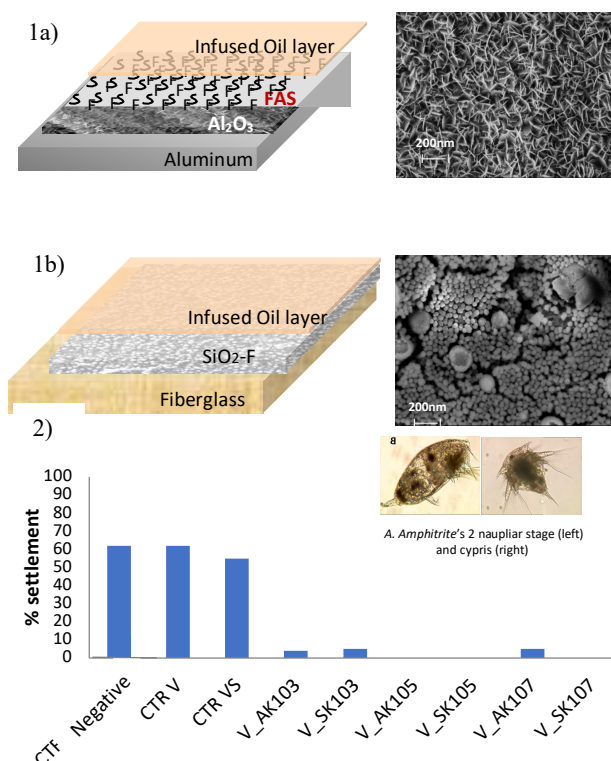
<sup>1</sup> CNR-ISTEC Institute of Science and Technology for Ceramic, Faenza, Italy

<sup>2</sup> CNR-IAS Institute for the Study of Anthropic Impact and Sustainability in the Marine Environment, Genoa, Italy

## Abstract:

Marine bio-fouling consists in undesirable settlement and accumulation of marine microorganism, plants and animals on submerged surface of materials and it has huge adverse influence on infrastructures. Marine bio-fouling increases the weight and roughness of ship hulls which raises the frictional resistance and causes additional fuel consumption [1]. The microorganisms contained in the sea water in contact with the surface of the hull adhere causing the growth of biofilm. Superhydrophobic surfaces have been investigated as a potential solution to avoid this phenomenon. We present the design of anti-wetting hybrid coating for antifouling environments (Figure 1a,b) that comprise an inorganic, porous layer based on functionalized ceramic nanoparticles ( $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{SiO}_2\text{-F}$ ) obtained via sol-gel, and an organic layer consisting of fluoralkylsilane molecules. Moreover, liquid-infused coatings were fabricated by infusion of fluorinated oil PFPE in the porous hybrid coating[2]. These coatings were deposited on materials like steel, aluminum and fiberglass. The reduction of the accretion of fouling was investigated by larval settlement test (*A. Amphrite*, a hard fouler[3]). In addition, release tests were carried out to evaluate the toxicity of the coating for the marine environment in accordance with the ASTM 6442 standard[4].

**Keywords:** hydrophobic coating/surface, ceramic nanoparticles, antifouling surface, bio-inspired coating for antifouling application



**Figure 1:** Figure illustrating the scheme of a)  $\text{SiO}_2\text{-F}$  nanoparticles coating infused in Kritox and the morphology of  $\text{SiO}_2\text{-F}$  nanoparticles coating. 1b) Scheme of  $\text{Al}_2\text{O}_3$  nanostructured coating with fluoroalkylsilane and infused Krytox and the morphology flower-like structure of  $\text{Al}_2\text{O}_3$  coating.

**Figure 2:** % of settlement of Amphrite larvae on different surfaces.

## References:

- Li, Y. & Ning, C. Latest research progress of marine microbiological corrosion and bio-fouling, and new approaches of marine anti-corrosion and anti-fouling. *Bioact. Mater.* **4**, 189–195 (2019).
- G.Boveri, A. Corozzi, F. Veronesi, M. Raimondo, *Coatings*, 2021;11,77
- Reddy G.K.K. Rajitha K. Nacharaiah Y.V. 2020 Antibiofouling potential of 1-alkyl-3-methylimidazolium ionic liquid: Studies against biofouling barnacle larvae. *Journal of Molecular Liquids*, 302, 112487.
- ASTM D6442-06 (2012)- Standard test method for determination of copper release fate from antifouling coatings in substrate ocean water. ASTM International

# Steam-corrosion resistance of polycrystalline Ag thin optical coatings improving by grain-boundary segregation effect

G. Marchii<sup>1</sup>, D. Samsonov<sup>1</sup>, I. Tereschenko<sup>1,2</sup>, A. Guba<sup>3,1</sup>, E. Mukhin<sup>1</sup>

<sup>1</sup> Plasma-Surface Interactions Lab, Ioffe Institute, St.-Petersburg, Russia

<sup>2</sup> Spectral-Tech, St.-Petersburg, Russia

<sup>3</sup> Institute of Chemistry, St.-Petersburg State University, Russia

## Abstract:

High and wideband reflectivity makes thin film Ag-based reflective coatings widespread in optical instruments engineering. Some challenging applications claim corrosion resistance, including that in high temperatures. Typical reason for silver corrosion is access of Cl, O and S ions from water solute or humid environment. Elevated temperature boosts this process.

Various protective coatings are designed to improve Ag optical coating corrosion resistance. They typically comprise one or more dielectric barrier layers used sometime in combination with an ultrathin (<1 nm) layer, which keeps the barrier layers better adhered to the silver<sup>1</sup>. Despite several authors showed a crucial role this layer plays in coating durability, simultaneous action of steam corrosion and thermal effects currently is not investigated.

Having compared corrosion features in samples with adhesion layers of CrN<sub>x</sub> and NiCrN<sub>x</sub>, *Folgnier et al* showed that presence of Ni in the adhesion layer determines the character of corrosion<sup>2</sup>. In the first sample, it takes place on Ag-CrN<sub>x</sub> interface, accompanied with significant depletion of silver layer. Corrosion features on the second sample demonstrate the opposite behavior: reactions occur mainly at the bottom of the silver layer affecting silver depletion.

The nature of this corrosion effect, as well as influence of the adhesion layer, remain unknown. Intergranular corrosion is suspected to make a large contribution to observed effects, since corrosion of long grain boundaries (GB) is faster than within bulk. Controlling the properties of polycrystalline thin films by direct modification of GB properties, is actively developed now. There are plenty of examples, showing the effect of GB segregation in immiscible alloys (solid solutes) improves thermal stability of nanomaterials by suppressing recrystallisation, GB migration and GB growth<sup>3</sup>. The effect of GB segregation on intergranular corrosion resistance, considered almost the only way for controlling film properties without drastic decrease in optical performance.

Ni was selected as a candidate element for GB segregation investigations, due to its known role in silver corrosion resistance improvement. Both experimental<sup>4,5</sup> and numerical<sup>6</sup> studies show, that Ni tends to segregate in silver. *Pan and Sansoz* report this effect can be used to improve silver strain resistance<sup>7</sup>.

The segregation influence on Ag coating corrosion resistance was studied by density functional theory method. Different GB configurations were investigated for Ni segregation in Ag by molecular dynamics with semi-empirical potential, suggested by *Pan et al.*<sup>6</sup>. The Ni GB segregation used for improving thermal stability of thin Ag films was studied as well. Impact of Ni presence on optical properties was also investigated.

An experimental technique being developed for quantitative segregation assessment was based on measuring the film electrical properties. This approach could become a promising non-destructive method for quality control of corrosion-resistant highly reflective coatings.

**Keywords:** Ag-based mirrors, reflective coatings, segregation, corrosion, nanomaterials

## References:

1. Wolfe, J. D., Laird, R. E., Carniglia, C. K. & Lehan, J. P. Durable silver-based antireflection coatings and enhanced mirrors. *Optical Interference Coatings* **17**, 115–117 (1995).
2. Folgnier, K. A. *et al.* Environmental durability of protected silver mirrors prepared by plasma beam sputtering. *Appl. Opt., AO* **56**, C75–C86 (2017).
3. Murdoch, H. A. & Schuh, C. A. Stability of binary nanocrystalline alloys against grain growth and phase separation. *Acta Materialia* **61**, 2121–2132 (2013).
4. Gas, P., Poize, S., Bernardini, J. & Cabane, F. A new approach to correlate grain boundary diffusion and segregation deduced from experimental measurements. *Acta Metallurgica* **37**, 17–24 (1989).
5. Surholt, T., Minkwitz, C. & Herzig, Chr. Nickel and selenium grain boundary solute diffusion and segregation in silver. *Acta Materialia* **46**, 1849–1859 (1998).

6. Pan, Z., Borovikov, V., Mendelev, M. I. & Sansoz, F. Development of a semi-empirical potential for simulation of Ni solute segregation into grain boundaries in Ag. *Modelling Simul. Mater. Sci. Eng.* **26**, 075004 (2018).
7. Pan, Z. & Sansoz, F. Heterogeneous solute segregation suppresses strain localization in nanocrystalline Ag-Ni alloys. *Acta Materialia* **200**, 91–100 (2020).

# The Effect of Carbon Nanotubes on the Corrosion Resistance of Waterborne Polyurethane-Acrylate Coatings

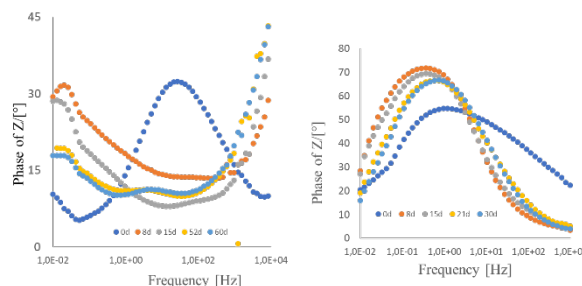
J. T. Corredor<sup>1\*</sup>, L.F. Giraldo<sup>1\*</sup>,

<sup>1</sup> Institute of Chemistry, Antioquia University, Medellín, Colombia

## Abstract:

Nowadays Hybrid waterborne polyurethane/acrylate (WPUA) dispersions are an important alternative as organic coating due to low volatile organic compounds, and excellent adhesion to metal. However, WPUA coating have poor water and weather resistance and their barrier properties must be improved so that one can be used as effective coatings against the corrosion degradation of steel structures. In recent years, it has been verified that the introduction of nanostructures as reinforcing agent is an effective way to improve the long-term corrosion protection. Carbon nanotubes (CNTs) are considered to be an attractive filler for polymer composites because of their high density, high thermal stability and high aspect ratio. We report the effect of the single wall and the multi wall CNTs content on the corrosion resistance of the WPUA coating in 3,5% NaCl at the room temperature, using electrochemical impedance spectroscopy (EIS). The waterborne polyurethane-acrylate dispersions ultrasonically mixed with MWCNTs and SWCNT at different contents of 0, 0.1 and 0.5 wt% were applied on low carbon steel substrates. The composites WPUA coatings with a thickness of about 12-15  $\mu\text{m}$  were prepared on the steel substrates with a drawdown rod. After 15 days of immersion in 3.5 wt% NaCl, the coating resistance of the WPUA coating was  $1.6 \times 10^3 \Omega \text{ cm}^2$ , whereas those of the composite coatings containing 0.1 SWCNTs and 0.1 wt% MWCNTs were  $2.0 \times 10^3$  and  $2.2 \times 10^3 \Omega \text{ cm}^2$ , respectively. Corrosion resistance decreased when CNT increase to 0.5 wt%.

**Keywords:** MWCNTs, SWCNT, waterborne polyurethane-acrylate, composite coatings, anti-corrosion coatings, EIS.



**Figure 1:** Bode phase plot of the WPUA and SWCNT/WPUA coatings with different immersion durations: (left panel) the pure WPU coating (right panel) MWCNT/WPUA coating.

0.1% MWCNT-WPUA exhibited the higher phase angle during 30 days of immersion as shown in Fig. 1. this prove its corrosion resistance during the whole immersion process. The results demonstrated the positive effect of the incorporation of low MWCNTs content on the anticorrosive ability of the coatings.

## References:

1. Song, D., Yin, Z. (2017) Effect of carbon nanotubes on the corrosion resistance of water-borne acrylic coatings, *Progress in Organic Coating.*, 110, 182-186.
2. Wang, F., Feng, L. (2018) Properties of Waterborne Polyurethane Conductive coating with Low MWCTs Content by Electrostatic Spraying, *Polymers.*, 10, 1406, 2-10.

# Adhesion of polyurethane coatings with powder fillers on composite substrates

Paulina Mayer<sup>1\*</sup>, Anna Dmitruk<sup>1</sup>, Marek Lubecki<sup>2</sup>, Kinga Rosochacka, Peter Kus<sup>3</sup>

<sup>1</sup>Wrocław University of Science and Technology, Department of Lightweight Element Engineering, Foundry and Automation, Faculty of Mechanical Engineering, St. Wyspiańskiego 27, 50-370 Wrocław, Poland; PL,

\*e-mail corresponding author: paulina.mayer@pwr.edu.pl

<sup>2</sup>Wrocław University of Science and Technology, Department of Maintenance and Operation of Logistics, Transportation and Hydraulic Systems, Faculty of Mechanical Engineering, Wyspiańskiego 27, 50-370 Wrocław, Poland; PL,

<sup>3</sup>Charles University, Faculty of Mathematics and Physics, Ke Karlovu 3, 121 16 Praha 2, Czech Republic

## Abstract:

The study presents the pull-off strength test results of polyurethane coatings with powder fillers. Polymer coatings with 20 % wt. powder fillers were applied to the substrates of three laminates reinforced with aramid, carbon or glass fabrics. The influence of adhesion of polyurethane coatings with was also investigated depending on the method of preparing the laminate surface. The layers were applied to three different surfaces of the laminates: immediately after the process of their production (infusion), after degreasing in acetone and after abrasive blasting. Before applying the coatings on composite substrates, their roughness was measured and the roughness parameters Ra and Rz were determined. Adhesion and cohesion detachment mechanisms were observed during the tests. The method of preparation and the type of composite substrate, as well as the type of powder filler used, affect the adhesion of the coatings.

The laminates used as substrates were made by the infusion method. It consisted in arranging the fabrics one after the other on a glass, smooth form. Then, in each separate production cycle, a synthetic peel-ply fabric was applied to the properly arranged layers of materials. The last layer - an infusion mesh made of polyethylene fibers - was placed on the previously prepared delamination, so that it would help to evenly distribute the resin over the entire mold area. The last step was to tightly cover the mold with a vacuum bag with the use of double-sided butyl tape and assembly on one side of the spiral pipe with which the resin was forced, and on the other side - a vacuum pump. After the infusion process, each laminate was cured for 8 hours at a temperature of 80°C. Huntsman's Araldite LY 1564 epoxy resin with Aradur 3487 amine hardener was used to produce laminates - in proportions by weight 100: 34, respectively. Araldite

LY 1564 is a substance characterized by a low viscosity (1200-1400 mPas) and a density of 1.1-1.2 g/cm<sup>3</sup>, intended for infusion method. The composition of the epoxy resin Araldite LY 1564 with the hardener Aradur 3487 is often used to supersaturate the composite layers due to its properties such as: no shrinkage during curing, odorlessness and hydrophobicity. As a result, 3 different types of laminates were obtained, namely:

G - 4 layers of glass emulsion mat (300 g) with 3 layers of glass fiber fabric 163 g/m<sup>2</sup> plain 1/1  
C - 4 layers of glass emulsion mat (300 g) with 3 layers of carbon fiber fabric 160 g/m<sup>2</sup> twill 2/2  
A - 4 layers of glass emulsion mat (300 g) with 3 layers of aramid fiber fabric 173 g/m<sup>2</sup> plain 1/1

Prepared, cut composite materials produced by the infusion method were as the basis for polymer coatings. The polymer coatings that were applied to the samples consisted of neat Sika F180 polyurethane and with the addition of four different fillers with a content of 20% by weight. The applied powder fillers were: Sillikoloid P87, Sillitin N85, Aktisil AM, Aktisil EM.

**Keywords:** adhesion, pull-off test, coating with fillers, composite substrates, laminates, roughness measurement

## References:

1. Paulina Mayer, Anna Dmitruk, Marta Jóskiewicz, Mateusz Głuch: Pull-off strength of fiber-reinforced composite polymer coatings on aluminum substrate, Journal of Adhesion. 2021, vol. 97, nr 15, s. 1371-1387.  
<http://dx.doi.org/10.1080/00218464.2020.1771556>

2. Paulina Mayer, Anna Dmitruk, Jacek Kaczmar: Adhesion of functional layers based on epoxy and polyurethane resins for aluminum substrate, International Journal of Adhesion and Adhesives. 2021, vol. 109, art. 102899, s. 1-6.  
<https://doi.org/10.1016/j.ijadhadh.2021.102899>
3. Paulina Mayer, Anna Dmitruk, Nicole Leja, Emilia Pakiet: Pull-off strength of fibre-reinforced composite polymer coatings on steel substrate, Journal of Adhesion. 2020, s. 1-19.  
<https://doi.org/10.1080/00218464.2020.1831478>



# Interactions of poly(ethyl-cyanoacrylate) with surfaces of aluminium oxide and titanium oxide

P. Moritz<sup>1,\*</sup>, L. Wegewitz<sup>1</sup>, W. Maus-Friedrichs<sup>1</sup>

<sup>1</sup> Clausthal Centre For Material Technology, Clausthal University of Technology, Clausthal-Zellerfeld, Germany

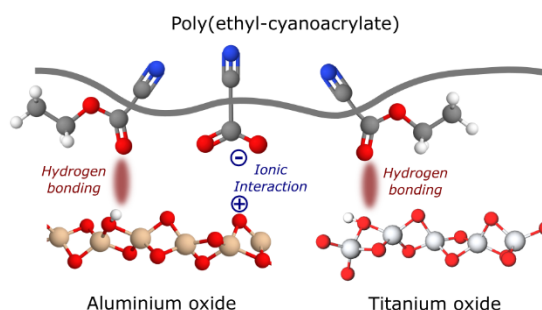
## Abstract:

Cyanoacrylates are highly reactive adhesives that can create a stable bond between different material surfaces within a few seconds. While the curing reaction has been intensively researched, the bonding mechanism is poorly understood. Previous studies investigated the interaction with model substrates such as Au and SiO<sub>2</sub> [1] or the interaction of chemically similar model polymers such as poly(methyl-methacrylate) [2]. The interactions with technically relevant oxidized metals have not yet been investigated.

In this study, we report the molecular interaction of poly(ethyl-cyanoacrylate) (PECA) with the surfaces of Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub>. Spin coating is used to deposit nm-thin films of the PECA onto the oxide surfaces, which allows the interface to be studied by X-Ray Photoelectron Spectroscopy (XPS). On both substrates, the adhesive interacts via hydrogen bonds through the free electron pairs of the carbonyl oxygen. On the Al<sub>2</sub>O<sub>3</sub>, there is also evidence of ionic interaction through the formation of a COO<sup>-</sup> group.

Further investigations using Metastable Induced Electron Spectroscopy (MIES) on very thin PECA films (< 2nm) allow conclusions to be drawn about the molecular orientation of the polymer. It is found that the PECA molecule is preferentially oriented with the carbonyl group towards the oxide surface. This is consistent with the hydrogen bonds found in the XPS results.

**Keywords:** adhesive, cyanoacrylate, thin films, spin coating, X-ray Photoelectron Spectroscopy, Metastable Induced Electron Spectroscopy



**Figure 1:** Figure illustrating the proposed interaction mechanism of poly(ethyl-cyanoacrylate) (PECA) with the oxide surfaces of Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub>.

## Acknowledgments:

The project was funded by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) – Project-ID 394563137 – SFB1368.

## References:

1. Moritz, P.; Bürger, F.; Höfft, O.; Wegewitz, L.; Maus-Friedrichs, W. (2021) Bonding Mechanism of Cyanoacrylates on SiO<sub>2</sub> and Au: Spectroscopic Studies of the Interface, *J. Phys. Chem. C.*, 125, 42, 23409-23417.
2. Watts, J. F.; Leadley, S. R.; Castle, J.E.; Blomfield, C. J. (2000), Adsorption of PMMA on oxidized Al and Si substrates: An investigation by high-resolution X-ray photoelectron spectroscopy, *Langmuir*, 16, 2292-2300.

## Light Activated Inorganic Agents - the next generation of photocatalytic paints

R. Bucureşteanu<sup>1</sup>, V. Chihaia<sup>2</sup>, B. Cojocaru<sup>3</sup>, M. Ioniţă<sup>4</sup>, A. Ficai<sup>5</sup>, L.M. Diţu<sup>1</sup>, G. Mihaescu<sup>1</sup>

<sup>1</sup> Department of Microbiology, Faculty of Biology, University of Bucharest, Bucharest, Romania

<sup>2</sup> Institute of Physical Chemistry "Ilie Murgulescu", Romanian Academy, Bucharest, Romania

<sup>3</sup> Department of Organic Chemistry, Biochemistry & Catalysis, Faculty of Chemistry, University of Bucharest, Bucharest, Romania

<sup>4</sup> Faculty of Applied Chemistry and Materials Science, University Politehnica of Bucharest, Bucharest, Romania.

<sup>5</sup> Department of Science and Engineering of Oxide Materials and Nanomaterials, Faculty of Applied Chemistry and Materials Science, University Politehnica of Bucharest, Bucharest, Romania

### Abstract:

To protect the surfaces from being contaminated with pathogens, antimicrobial coatings have been proposed and applied in the form of paints or polymeric layers containing biocidal agents, usually represented by 1,2-Benzothiazol-3 (2H)-one type, Triclosan, various polymers, with copper or silver incorporated. A major problem with these products is the phenomenon of sublimation of the biocidal active agent, leading to a decrease in the active concentration and the loss of their protective properties. In this context, LAIA (Light Activated Inorganic Agents) composed of a mix of metallic Cu, Cu<sub>2</sub>O, and CuO is an innovative concept of functional dual catalytic composite which is defined as photocatalytic reaction though which generates in the same time catalytic reaction in the absence of light. The LAIA product was characterized by X-ray diffraction (XRD) and X-ray energy-dispersive spectroscopy (XEDS), XPS under controlled environmental conditions, transmission electron microscopy (TEM, Brunauer-Emmett-Teller (BET) nitrogen adsorption/desorption isotherms and pore size distribution. The antimicrobial activity was evaluated using qualitative and quantitative standard methods (adapted CLSI method and NF EN 1276: 2019 Standard). The microbial strains were represented by *Pseudomonas aeruginosa* (CECT 116 = ATCC 15442), *Escherichia coli* (CECT 405 = ATCC 10536), *Salmonella enterica* subsp. *enterica* serovar. *typhimurium* (CECT 443), *Klebsiella pneumoniae* (CECT 8453), *Staphylococcus aureus* (CECT 239 = ATCC 6538), *Streptococcus pyogenes* (ATCC 19615), *Enterococcus faecium* VRE (ATCC 700221), *Enterococcus hirae* (CECT 4081 = ATCC 10541), *Candida albicans* (CECT 1394 = ATCC 10231). LAIAs photocatalytic sample under analysis shows photocatalytic activity

demonstrated by discoloration of an aqueous solution of Methylene Blue with a concentration of 20 mg/l both for irradiation exclusively with light from the proximate UV range and for irradiation exclusively with light from the visible range. High-energy-resolution analysis of the Cu 2p region clearly indicated that the particle surfaces were mostly Cu(II). XPS wide-energy survey spectra collected on these samples showed some unexpected zirconium peaks. Cu<sub>2</sub>O (copper (I) oxide; cuprous oxide) is a red powder and can also be produced as nanoparticles. Similar activity to CuO (copper(II) oxide; cupric oxide) has been shown. XPS wide-energy survey spectra collected on these samples showed some unexpected zirconium peaks. The product LAIAS – light activated inorganic agents, batch 520/2021, when is diluted at 50% and 10% (w:v) in sterile distilled water, shows bactericidal activity after 60 minutes at 20°C ± 1°C, under clean conditions (bovine serum albumin 0.3 g/L).

**Keywords:** antimicrobial coatings, Light Activated Inorganic Agents, biomedical applications.

### References:

1. Bucureşteanu R., Diţu L.M., Ionita M., Calinescu I., Raditoiu V., Cojocaru B., Cinteza O., Curutiu C., Holban A.M., Enachescu M., Enache L., Mustatea G., Chihaia V., Nicolaev A., Borcan E.L., Mihaescu G. Preliminary Study on Light-Activated Antimicrobial Agents as Photocatalytic Method for Protection of Surfaces with Increased Risk of infections. *Materials* 2021, 14, 5307. <https://doi.org/10.3390/ma14185307>.

**Acknowledgments:** We gratefully acknowledge the financial support offered by the research grant no. 527/2020, project code: PN-III-P2-2.1-PED-2019-1825, UEFISCDI.

# Colloidal and Thermal stability of Hybrid Waterborne Polyurethane Dispersions Prepared by Grafting Method

Luis F. Giraldo\*, Jeaneth T. Corredor, Esneyder Ruiz, Victor H. Orozco

<sup>1</sup> Laboratorio de Investigacion en Polímeros (LIPOL), Instituto de Química, Universidad de Antioquia, Medellín, Colombia

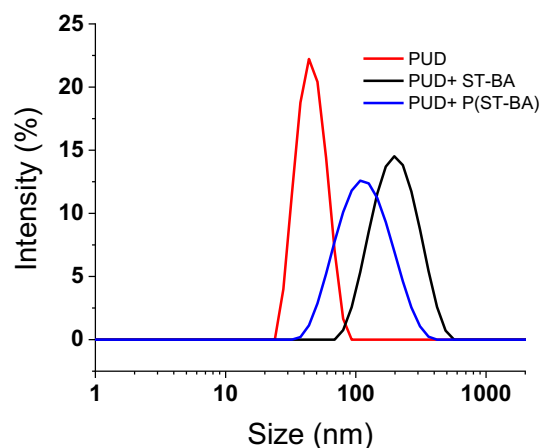
## Abstract:

Waterborne polyurethane dispersion (PUD) continues being one of the materials more promissory to replace solvent-based polyurethane systems used in the coating and adhesive industry. Due to environmental restrictions, most formulations that use volatile organic compounds must be changed to their water base counterpart. Even though the coatings made with polyurethane show good tensile strength, gloss, and resilience, improvements in the thermal and tribological properties have to be done. In this sense hybrid waterborne polyurethane dispersions have emerged as a promising alternative not only to improve physicochemical properties such as scratch resistance, tensile stress, and hydrophobicity but also to make these polyurethanes-based production processes more economical<sup>1</sup>. The hybridization of polyurethanes can be done with vinyl-acrylic polymers by means of interpenetrating networks, end-capping or grafting from the process, in the last case a heterobifunctional monomer with dihydroxyl groups and double bonds are used as crosslinking agent between the polyurethane and acrylic polymeric chains. Our research group reported for the first time the bisphenol-a-glycidyl methacrylate (Bis-GMA) as a chain extender of polyurethane and crosslinking agent<sup>2</sup>. In this work, we report the use of Bis-GMA to get an HPUD by means of grafting from the process reacting the monomer during the pre-polymer polyurethane formation

Figure 1 shows the particles size distribution of aqueous polyurethane dispersion before and after styrene (ST) and butyl acrylate (BA) incorporation, as well as the particle size distribution after polymerization of acrylic monomers. Table 1 summarizes the colloidal properties. The PUD has a mean particle size of 44 nm, when the styrene and butyl acrylate are incorporated the mean particle size increase to 168 nm, which indicates that the acrylic monomers are incorporated into the PU nanoparticles swelling them. Once the styrene and butyl acrylate are polymerized the mean particles size decrease again obtaining a final mean particle size of 106 nm. This behavior is expected due to the change from Van der Waals interaction to covalent bonds in the P(ST-

BA) after polymerization. The zeta potential is higher for the HPU compared to PU with a value of -73 mV which suggests greater colloidal stability. The polymers were characterized by GPC, FTIR and the water contact angle was measured on the polymer films as well as TGA and DSC. The hybrid polymer shows a higher molecular weight, thermal stability, and a more hydrophobic film.

**Keywords:** Waterborne polyurethane dispersion, Interpenetrating polymer network, styrene, and butyl acrylate, coating, and adhesives.



**Figure 1.** Particle size distribution of PUD and HPUD with ST and BA before and after polymerization, PU+P(ST-BA).

## References:

- Ospina, A. C. *et al.* Study of waterborne polyurethane materials under aging treatments. Effect of the soft segment length. *Prog. Org. Coatings* **138**, (2020).
- Alvarez, G. A., Fuensanta, M., Orozco, V. H., Giraldo, L. F. & Martín-Martínez, J. M. Hybrid waterborne polyurethane/acrylate dispersion synthesized with bisphenol A-glycidylmethacrylate (Bis-GMA) grafting agent. *Prog. Org. Coatings* **118**, 30–39 (2018).

# Antibacterial SnO<sub>2</sub> coatings applied on Ti6Al7Nb alloy for applications in bone surgery

M. Basiaga<sup>1,\*</sup>, W. Walke<sup>1</sup>, J. Lison<sup>1</sup>

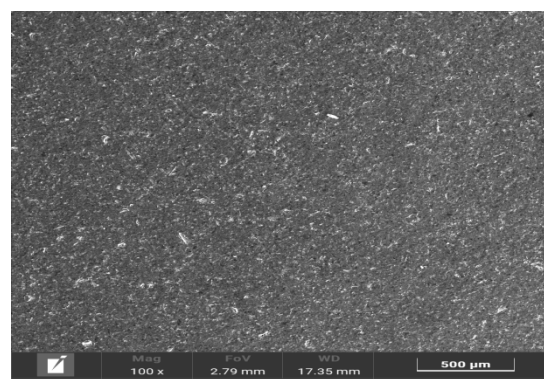
<sup>1</sup> Faculty of Biomedical Engineering, Silesian University of Technology, Zabrze, Poland

## Abstract:

Immediately after inserting the implant into the body, its surface is wetted by body fluids and then coated with serum or extracellular matrix proteins such as fibrinogen, fibronectin, albumin and others. The presence of these proteins is necessary to initiate the process of adhesion of stem cells and their subsequent differentiation into osteoblasts, and thus the process of osseointegration. One of the main factors disturbing the osseointegration process is the colonization of the surface of the biomaterial by bacteria initiating the development of the infection, which is creating bacterial biofilm. Reducing the risk of biofilm development on the surface of biomaterials is one of the main directions of research development that allows for effective reduction of biofilm related infection. The main approaches have involved modifying the surface of biomaterials to reduce microbial adhesion and interfering with the initial phases of biofilm development [1, 2]. The conducted research is aimed at developing materials resistant to colonization and at the same time it is safe for the human body. Despite the involvement of many research centers in the world, the issue of coatings with antibacterial properties is based on partial research. There are no studies comprehensively synthesizing physicochemical and microbiological issues in relation to the specificity of the skeletal system. Against this background, the concept of work was born by modifying the Ti6Al7Nb alloy with the SnO<sub>2</sub> coating using the ALD method. As part of the assessment of the suitability of such a proposed surface modification, pitting corrosion resistance tests, SnO<sub>2</sub> coating adhesion tests to the substrate, abrasion resistance tests, surface wettability tests, surface topography tests (fig.1), microstructure tests and bacteria *S aureus* and *E coli* adhesion tests were carried out. Coli to the modified surface of the Ti6Al7Nb alloy. Firstly, the surface of the Ti6Al7Nb alloy was subjected to both sandblasting and polishing. Then, SnO<sub>2</sub> was applied to the prepared surface by the ALD method with changing parameters of the deposition process. The variable parameters were temperature and the number of cycles. On the basis of the obtained data, different physicochemical

properties of SnO<sub>2</sub> coatings depending on the number of cycles and the temperature of application were found. It was found that the applied coating had a beneficial effect on the antibacterial properties, with the apparently smaller number of bacterial colonies compared to the substrate without the coating. The knowledge gained on this basis is of practical importance for the application of this type of surface modification for various types of implants used in the skeletal system.

**Keywords:** SnO<sub>2</sub> layer, ALD method, Ti6Al7Nb alloy, antibacterial layers, electrochemical properties, mechanical properties, biological properties, biomedical applications.



**Figure 1:** Surface morphology of Ti6Al7Nb alloy with SnO<sub>2</sub> coating,

## Acknowledgements:

The project was funded by the National Science Centre, Poland allocated on the basis of the decision No. 2018/29/B/ST8/02314

## References:

1. Bryers J.D.(2008) Medical Biofilms, *Biotechnol Bioeng*, 1, 1-18.
2. Lewis K. (2001) Riddle of biofilm resistance. *Antimicrob Agents Chemother*, 45, 999-1007.

# Ti13Zr13Nb alloy modified of tin dioxide by means of nanoPVD and ALD methods for an implant used in the skeletal system

W Walke<sup>1,\*</sup>, M. Basiaga<sup>1</sup>, K. Goldsztajn<sup>1</sup>

<sup>1</sup> Faculty of Biomedical Engineering, Silesian University of Technology, Zabrze, Poland

## Abstract:

In recent years, the Ti13Zr13Nb alloy, among the more than forty alloys investigated to date, seems to be the best candidate due to its non-toxicity and relatively low Young's modulus [1]. Nb and Zr are stabilizers of  $\beta$ -phase and cause a decrease of the Young's modulus so that the titanium Ti13Zr13Nb alloy is more biocompatible than others. Moreover, the stable oxide TiO<sub>2</sub> film 1–4 nm thick is spontaneously formed on the surface the titanium Ti13Zr13Nb alloy. Its ability to natural self-passivation and presence of the titanium dioxide on the surface reduces the risk of migration of metal ions to body fluids after implantation [2]. The weak bonding of titanium and its alloys to bone and its lack of bioactivity have resulted in a great variety of different surface modification techniques. The surface modification techniques perform very important function in shaping the physicochemical properties of the Ti13Zr13Nb alloy intended for implants. The methods of modifying the outer layers must ensure repeatability and uniformity of their physical and chemical properties. The structure and chemical composition of the layer of implants made of Ti13Zr13Nb alloy can be modified using various methods, among which the anodic oxidation and sol-gel dominate [1, 2]. One of the most important aspects of miniaturized implants is to provide them with a constant geometric feature along their entire length, which the above-mentioned modifications are not able to provide. Therefore, in this context, the most suitable surface modification technique is the application of layers using the ALD (Atomic Layer Deposition) and nanoPVD (Physical Vapor Deposition) methods. Apart from the improvement of biocompatibility, an important issue related to the production of layers is also the appropriate set of their physicochemical properties. For that reason, the study attempts to evaluate the physical and chemical properties of the applied SnO<sub>2</sub> layers on the Ti13Zr13Nb alloy substrate using both the ALD and nanoPVD methods. As part of the evaluation of electrochemical properties of the surface layers formed in this way, tests of resistance to pitting and crevice corrosion were carried out,

as well as tests with the use of electrochemical impedance spectroscopy. Ultra-nanohardness, adhesion to the metal substrate (scratch test) and resistance to abrasion (pin-on-disc) studies were performed to determine the mechanical properties of applied layers. Additionally, tests of the surface topography and its wettability were carried out. Moreover, the thickness measurements of the analyzed layers were performed and estimated with the use of the ellipsometric method. Based on the obtained results, different physicochemical properties depending on the applied technology of application were found. Proposing suitable variants of surface treatment using the ALD and nanoPVD methods has perspective importance and will contribute to the development of technological conditions with precise parameters for the production of oxide coatings on implants used in bone surgery.

**Keywords:** SnO<sub>2</sub> layer, ALD method, PVD method, Ti13Zr13Nb alloy, electrochemical properties, mechanical properties, skeletal system.

## Acknowledgements

The project was funded by the National Science Centre, Poland allocated on the basis of the decision No. 2018/29/B/ST8/02314

## References:

1. Zielinski A, Sobieszczyk S., Seramak T., Serbinski W., Swieczko-Zurek B., Osowska A (2011) Biocompatibility and bioactivity of load-bearing metallic implants *Adv. Mater. Sci.*, 10, 21-31,
2. Szklarska M., Dercz G, Simka W., Łosiewicz B. (2014) A.c. impedance study on the interfacial properties of passivated Ti13Zr13Nb alloy in physiological saline solution *Surf. Interface Anal.*, 46 pp. 698-701,

## Optimization of laser parameters for the injection moulds cleaning

R. Santos<sup>1</sup>, I. Marcelino<sup>2</sup>, T. Ferreira<sup>2</sup>, L. Pereira<sup>3</sup>, F.M. Costa<sup>1</sup>, N. M. Ferreira<sup>1</sup>

<sup>1</sup>i3N & Physics Department, University of Aveiro, Aveiro, Portugal

<sup>2</sup> Instituto Pedro Nunes, Coimbra, Portugal

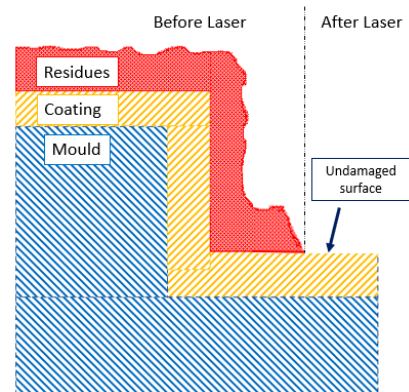
<sup>3</sup> MicroPlásticos, Figueira da Foz, Portugal

### Abstract:

In the plastic/polymers injection, the moulds must be regularly cleaned to avoid the residues accumulation on their surface, otherwise, the dimensions of the pieces produced are compromised.

Several examples of using lasers to clean moulds could be found. Such as using a CO<sub>2</sub> laser to clean the moulds in the glass industry or the laser fibre in the elastomers industry. The major difficulties to implement such technology is when the mould surface and the residues interact in the same manner with the laser beam used. A study of the implementation of the laser technology to clean the metallic mould of the polymer injection was addressed (Fig 1). The residues are polymeric and can be removed by ablation, using 1064nm radiation. The radiation is also highly absorbed by the mould surface and its coating. The optimization of the laser parameters such as power, frequency, scan speed, etc, is required for efficient removal of the residues without the degradation of the mould surface coating. In addition, the moulds studied are used to produce a complex piece, presenting small areas at different heights. The present study report also the optimization of mould division in different heights, like a topographic map (figure 2). The idea is to focus the laser beam on each plane with the same height, and proceed to clean it, rapidly and efficiently. The results obtained show the efficiency of laser technology in cleaning these moulds.

**Keywords:** Fiber Laser; clean moulds; injection moulds; topographic maps of the moulds.



**Figure 1:** Figure illustration of the methodology to clean the mould, using laser technology.



**Figure 2:** Topographic map of one part of the mould, where the different colours represent different heights planes.

### References:

1. <https://www.p-laser.com/glass-mould-cleaning>
2. <https://cleanmould.web.ua.pt/wp>
3. D. Höche, J. Kaspar, P. Schaaf, Laser Surface Engineering. Elsevier, 2015.
4. <https://www.ctborracha.com/limpeza->



# Design of polymeric coatings based on pH-sensitive acrylic terpolymers with potential application in the treatment of colonic diseases.

L.M Suárez Giraldo, V. Orozco López, L.F Giraldo Morales

Antioquía, Universidad de Antioquía, Medellín, Colombia

## Abstract

Oral drug delivery is one of the best alternatives to incorporate a drug into the body due to its simplicity, convenience, low cost, and safety. However, it is limited, if the target is after the stomach, by factors in the digestive environment such as enzymatic degradation, pH, solubility, among others[1]. For this reason, the development of effective drug delivery systems based on nanocarriers coated with pH-sensitive polymers improve the absorption and bioavailability of the drug in the body [2]. The design of these polymeric coatings is based on the versatility that can be conferred to certain polymers to modulate their properties such as molecular weight, hydrophobic/hydrophilic domains, adhesion, and their solubility dependence on pH, which allows modulating the delivery of active pharmaceutical ingredients (API) in a colonic environment[3].

Several organic and inorganic materials have been reported in the literature for applications and formulation strategies such as hydrogels based on synthetic and natural materials, microspheres, and nanoparticles. Among the materials used, polymers of synthetic origin such as copolymers based on acrylic acids and esters, whose physicochemical properties vary due to the presence of anionic, cationic, or neutral groups are highly promising as they allow the API in its solid dosage form to act during passage through the organism, this is due to the fact that these polymeric materials have better biocompatibility, biodegradability and mucoadhesive properties [4].

Based on the above, this seminar will analyze some previous results obtained in the development of a coating system based on a poly(methyl acrylate-co-methyl methacrylate-co-methacrylic acid) terpolymer, from the modulation of the proportions of the acrylic monomers, and their effect on the particle size distribution and z-potential, as well as the effect of the chemical structure on the controlled release of the API in the application of a static in vitro digestion model where the polymer coats HPMC (Hydroxypropylmethylcellulose)

capsules to determine the enteric functionalities, the encapsulation efficiency in a test molecule, all this is to obtain a drug delivery system with potential application in colonic diseases.

**Keywords:** Coatings, pH sensitive polymers, oral administration, colon diseases, HPMC Capsules, Dipping, acrylic polymers, active pharmaceutical ingredients (API).

## References

1. L. Liu, W. D. Yao, Y. F. Rao, X. Y. Lu, and J. Q. Gao, "pH-responsive carriers for oral drug delivery: Challenges and opportunities of current platforms," *Drug Deliv.*, vol. 24, no. 1, pp. 569–581, 2017, doi: 10.1080/10717544.2017.1279238.
2. A. Gonçalves, B. N. Estevinho, and F. Rocha, "Methodologies for simulation of gastrointestinal digestion of different controlled delivery systems and further uptake of encapsulated bioactive compounds," *Trends Food Sci. Technol.*, vol. 114, no. May, pp. 510–520, 2021, doi: 10.1016/j.tifs.2021.06.007.
3. Z. Shi, Q. Li, and L. Mei, "pH-Sensitive nanoscale materials as robust drug delivery systems for cancer therapy," *Chinese Chem. Lett.*, vol. 31, no. 6, pp. 1345–1356, 2020, doi: 10.1016/j.cclet.2020.03.001.
4. C. N. Patra, R. Priya, S. Swain, G. Kumar Jena, K. C. Panigrahi, and D. Ghose, "Pharmaceutical significance of Eudragit: A review," *Futur. J. Pharm. Sci.*, vol. 3, no. 1, pp. 33–45, 2017, doi: 10.1016/j.fjps.2017.02.001.

# Enhancing sensitivity and reducing unspecific adsorption in fiber-attached polymer hydrogels

A. Luongo<sup>1,2</sup>, T. Brandstetter<sup>1,2</sup>, J. R  he<sup>1,2</sup>

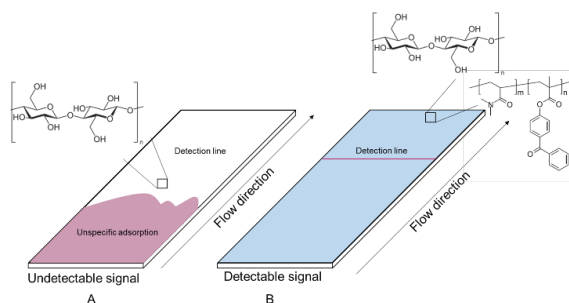
<sup>1</sup> Laboratory for Chemistry and Physics of Interfaces, Department of Microsystems Engineering (IMTEK), University of Freiburg, Georges-K  hler-Allee 103, 79110 Freiburg, Germany

<sup>2</sup> Freiburg Institute for Interactive Materials and Bioinspired Technologies (FIT), Albert-Ludwigs Universit  t Freiburg, Georges-K  hler-Allee 105, 79110 Freiburg, Germany.

## Abstract:

Cellulose is one of the most abundant biodegradable polymer in nature and it is the base material for the formation of all papers. They are hydrophilic and strongly porous; in addition, their fibrous structure allows fluids to be passively transported by capillary action, making this material a perfect candidate for pump-free microfluidics. Despite all the advantageous properties, paper-based devices, with very few exceptions, have been used on a rather limited scale in diagnostics and analytics. For applications with strongly abundant molecules, the sensitivity is sufficient, but for many others, light scattering and unspecific adsorption of analyte molecules on the device present major problems.<sup>1</sup> In this research project, coatings of papers with PDMAA-co-MABP via C,H-insertion reactions have been successfully performed, in order to reduce the unspecific adsorption. In a swollen hydrogel network, proteins are indeed unable to penetrate because of "entropic shielding", i.e. the significant loss of entropy induced by the penetration of macromolecules stretches further the hydrogel. This minimize the non-specific adsorption and accordingly retention of an analyte (e.g. protein) transported through the channel by capillary action. Moreover, while strong light scattering is detrimental for transmission through the paper, for fluorescence detection, strong light scattering even improves the net read-out signal because scattering enhances the excitation efficiency of fluorescent dyes (and their emission efficiency to the detector) in deeper layers of the paper and thus makes more molecules in the paper available for analysis.

**Keywords:** Lateral flow, cellulose, scattering, detection,  $\mu$ PAD, fluorescence, rapid diagnostic test.



**Figure 1:**

(A) Unspecific protein adsorption on a paper-based device is obtained, preventing the detection of any signal. (B) When the paper is polymer-modified, protein repellency allows the flowing of the target molecules and eventually the signal detection.

## References:

1. Hu, J.; Wang, S. Q.; Wang, L.; Li, F.; Pingguan-Murphy, B.; Lu, T. J.; Xu, F. Advances in Paper-Based Point-of-Care Diagnostics. *Biosens. Bioelectron.* **2014**, *54*, 585–597.
2. Eyer, K.; Root, K.; Robinson, T.; Dittrich, P. S. A Simple Low-Cost Method to Enhance Luminescence and Fluorescence Signals in PDMS-Based Microfluidic Devices. *RSC Adv.* **2015**, *5* (17), 12511–12516.
3. Prucker, O.; Brandstetter, T.; R  he, J. Surface-Attached Hydrogel Coatings via C,H-Insertion Crosslinking for Biomedical and Bioanalytical Applications (Review). *Biointerphases* **2018**, *13* (1), 010801.

# The Influence of Yarn Fineness in Layer Interchanging Double Cloth on Woven Fabric Roughness

I. Schwarz\*, T. Badrov, S. Kovačević

University of Zagreb, Faculty of Textile Technology, Zagreb, Croatia

## Abstract:

All woven fabric surfaces consist of various types of imperfections and irregularities that are affected by the construction characteristics of the fabric, mostly weave and yarn fineness. Tactile property is a multidimensional concept consisting of several characteristics, among which is surface roughness, as the fundamental and most effective factor whose parameters along with the parameters of surface waviness describe the topographic woven fabric properties. Fabric topography is expressed by periodic repetition of waves in the vertical and horizontal directions caused by its structure. Surface roughness is the totality of microgeometric irregularities on the fabric surface and has a significant role in the final use of fabrics, especially in ones that are in contact with human skin. In order to establish the connection between fabric construction parameters and their roughness, it is sufficient to describe its surface properties at the meso level. For the purpose of this research, woven fabric with complex weave construction (layer interchanging double cloth) was designed, using different raw material fibers (AR - 95% Meta Aramid Conex NEO; 5% Para Aramid Twaron raw and MAC/CO - 45% Cotton Long Stapel Combed, 55% Modacrylic Sevel FRSA/L), which will provide a diversity of structure and thus various properties on the front and backside of the fabric. For this reason, an investigation on the properties of roughness has been conducted on the backside of woven fabrics, where MAC/CO yarn predominates with the purpose to obtain fabric comfort when in contact with the skin.

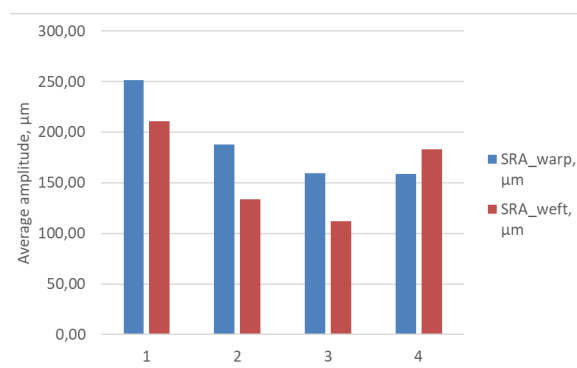
Figure 1 shows a correlation between the average amplitude from the roughness profile with yarn fineness and fabric density. The mean amplitude of roughness profile, in weft, direction decreases with increasing yarn fineness in weft in the first three samples where the differences in weft density are negligible. The sample with the finest weft (Sample 4, 12.5x2 tex) has higher values of mean amplitude in weft, which is caused by the increased weft density (66 threads/cm), while the other samples had a slight difference in weft density (58 threads/cm). Regarding the mean amplitude for warp, whose density is the same for all

samples, the values decrease nonlinearly with increasing weft fineness. Statistical analysis confirmed that weft fineness has the greatest influence on the average wavelength and amplitude of the roughness profile (both  $R^2 = 0.91$ ), in warp direction.

**Table 1:** Woven fabric specifications

Sample	Warp	Tt (tex)	Weft 1	Tt (tex)	Weft 2	Tt (tex)
1	AR	17x2	AR	17x2	MAC/CO	20x2
2	AR	17x2	AR	17x2	MAC/CO	17x2
3	AR	17x2	AR	17x2	MAC/CO	14x2
4	AR	17x2	AR	17x2	MAC/CO	12,5x2

**Keywords:** woven fabric, roughness, Fabric Touch Tester, layer interchanging double cloth, yarn fineness



**Figure 1:** The average amplitude of roughness profile of the tested samples

**Acknowledgment:** This research has been fully supported by the Croatian Science Foundation under the project Multifunctional woven composites for thermal protective clothing, number IP-2018-01-3170.

## References:

- Calvimontes, A., et al. (2010) Effects of Topographic Structure on Wettability of Differently Woven Fabrics, *Woven Fabric Engineering*, ed. Dobnik Dubrovski, P., Sciyo, Croatia, 71-92.
- Beyene, K.A. and Kumelachew, D.M. (2021) An investigation of the effects of weave types on surface roughness of woven fabric, *Text. Res. J.*, 0(0).

# Woven Fabric Roughness Conditioned by Weave

S. Brnada\*, A. Kalazić, S. Sabljak, T. Kaurin

University of Zagreb, Faculty of Textile Technology, Zagreb, Croatia

## Abstract:

The woven fabric structure is defined by the construction parameters, of which the most influential are weave and thread density. In order to characterize the woven fabric surface in detail, it is necessary to perceive it on several levels (macro, meso, micro). At the meso level, the topographic properties of woven fabrics are observed, which are described by the parameters of surface roughness and waviness. Typical topography is expressed by periodic repetition of waves in the vertical and horizontal directions resulting from its structure. Roughness is defined by closely spaced irregularities while waviness is manifested in widely spaced irregularities on the fabric surface. Different methods (contact and non-contact) have been developed to characterize the fabric surface roughness. For this research, the contact method on the Fabric Touch Tester device was used. The aim of this research is to present a detailed investigation and explanation of the effects of weave types on the surface roughness of woven fabrics in the warp and weft direction. Both directions consist of the same raw material (PA6.6/CO - 50% Polyamide 6.6, 50% Cotton) and the same fineness (15.5x2 tex). Fabric specification is shown in Table 1.

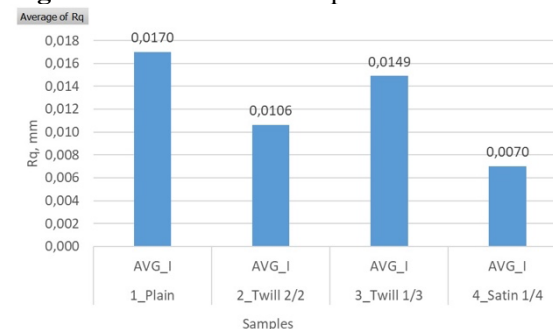
The root mean square (Figure 1) is the square root of the average of the square of the deviation of the profile from the mean line which represents the surface roughness with amplitude variations included. Figure 1 shows that the highest roughness has the fabric woven in plain weave while satin fabric has the lowest. This confirms the theory of the influence of thread flotation on the woven fabric surface roughness. In addition, in the case of twill fabrics, Twill 1/3 has a higher roughness due to full interlacement of every 4th thread. Figure 2 shows the height of the highest peak (Rp) and the depth of the deepest valley (Rv) in the roughness profile. The results are consistent with the ones shown in Figure 1. Fabric woven in Twill 1/3 shows an axis shift in favor of the highest peak (Rp) relative to the deepest valley (Rv).

**Table 1:** Woven fabric specifications

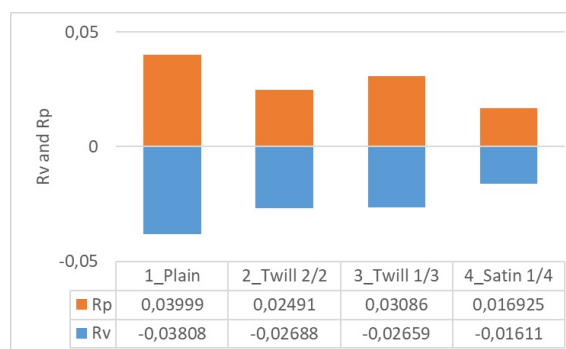
Sample_Weave	Warp density (thread/cm)	Weft density (thread/cm)
1 Plain	30	24
2 Twill 2/2	30	26
3 Twill 1/3	30	26
4 Satin 1/4	30	29

**Keywords:** surface roughness, woven fabric, weave, Fabric Touch Tester

**Figure 1:** The root mean square



**Figure 2:** Roughness profile extremes



**Acknowledgment:** This research has been supported by the European Union from the European Structural and Investment Funds, Operational programme Competitiveness and Cohesion, the European Regional Development Fund [Project Number KK.01.2.1.02.0064; Development of multifunctional non-flammable fabric for dual use].

## References:

1. Ezazshahabi, N., et all. (2015) Surface roughness assessment of woven fabrics using fringe projection moiré techniques, *Fibres Text. East. Eur.*, 23, 3(111).
2. Asghari Mooneghi, S., et all. (2014) Surface Roughness Evaluation of Textile Fabrics: A Literature Review, *Journal of Engineered Fibers and Fabrics*, 9(2).

# Plasma vortices in the near Earth environment

Khatuna Elbakidze

Iv. Javakhishvili Tbilisi State University, 0128 Tbilisi, Georgia

## Abstract:

Evolution of internal-gravity wave (IGW) structures in the dissipative ionosphere in the presence of a nonuniform zonal wind (a sheared flow) is investigated. The efficiency of the linear amplification of IGW structures in their interaction with a nonuniform zonal wind is analyzed. When there are sheared flows, the operators of linear problems are non-self-conjugate and the corresponding eigenfunctions are nonorthogonal, so the canonical modal approach is poorly suited for studying such motions and it is necessary to utilize the so-called nonmodal mathematical analysis. It is shown that, in the linear evolutionary stage, IGW efficiently extract energy from the sheared flow, thereby substantially increasing their amplitude and, accordingly, energy (by several orders). As the shear instability develops and the perturbation amplitude grows, a nonlinear self-localization mechanism comes into play and the process ends with the self-organization of nonlinear, highly localized, solitary IG vortex structures. The system thus acquires a new degree of freedom, thereby providing a new way for the perturbation to evolve in a medium with a sheared flow. Depending on the shape of the sheared flow velocity profile, nonlinear structures can be either purely monopole vortices or vortex streets against the background of the zonal wind. The accumulation of such vortices can lead to a strongly turbulent state in an ionospheric medium.

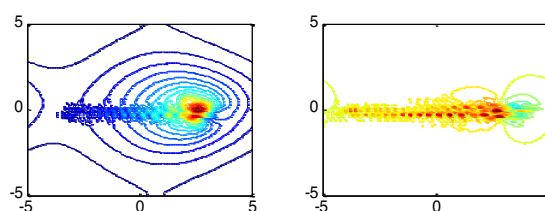
The Plasma vortices are often detected by spacecraft in the geospace (atmosphere, ionosphere, magnetosphere) environment, for instance in the magnetosheath and in the magnetotail region. Large scale vortices may correspond to the injection scale of turbulence, so that understanding their origin is important for understanding the energy transfer processes in the geospace environment. In a recent work, the THEMIS mission has detected vortices in the magnetotail in association with the strong velocity shear of a substorm plasma flow (which have conjugate vortices in the ionosphere. By analyzing the THEMIS data for that event, we find that several vortices in the magnetotail can be detected together with the main one, and that the vortices indeed constitute a vortex chain. Such vortices can cause the strong turbulent state in the different media. The strong

magnetic turbulence is investigated in the ionosphere as an ensemble of such strongly localized (weakly interacting) vortices.

## Acknowledgement:

The work is done by the grant FR17\_279 of Shota Rustaveli Georgian National Science Foundation.

**Keywords:** Turbulent State, Zonal Flow, Shear Flow, Mutual Transformation, Vortex Structures.



**Figure 1:** The figure represents evolution of initial monopole and random perturbations of the plasma flow, localized in a circle of radius 0.2 and magnetic field in a Gaussian inhomogeneous flows.

## References:

1. Aburjania G., Chagazia Kh. Self-organization of ULF large-scale electromagnetic wave structures in E region of the ionosphere at interaction with inhomogeneous zonal winds. *Plasma Phys. Rep.*, V. 37, N 2, P. 199-213. 2011.
2. Aburjania G., Khantadze A., Kharshiladze O. Nonlinear planetary electromagnetic vortex structures in F region of the ionosphere. *Plasma Phys. Rep.* V. 28, N 7, P. 633-638. 2002.
3. Akasofu, S.-I. *Physics of Magnetospheric Substorms*, D. Reidel, Dordrecht, Netherlands. 1976
4. Angelopoulos V. The THEMIS Mission. *Space Sci Rev* (2008) 141: 5–34, DOI 10.1007/s11214-008-9336-1. 2008.
5. Angelopoulos, V., et al. Multipoint analysis of a bursty bulk flow event on April 11, 1985, *J. Geophys. Res.*, 101, 4967–4990, doi:10.1029/95JA02722. 1996



# A plasma diagnostic package for spacecrafts with electric propulsion systems

V. Schneider<sup>1</sup>, T. Trottenberg<sup>1</sup>, H. Kersten<sup>1</sup>, J. Laube<sup>2</sup>, H. Henkel<sup>3</sup>, R. Wimmer-Schweingruber<sup>1</sup>

<sup>1</sup>Institute of Experimental and Applied Physics, Christian-Albrechts-University Kiel, Germany

<sup>2</sup>OHB System AG, Bremen, Germany

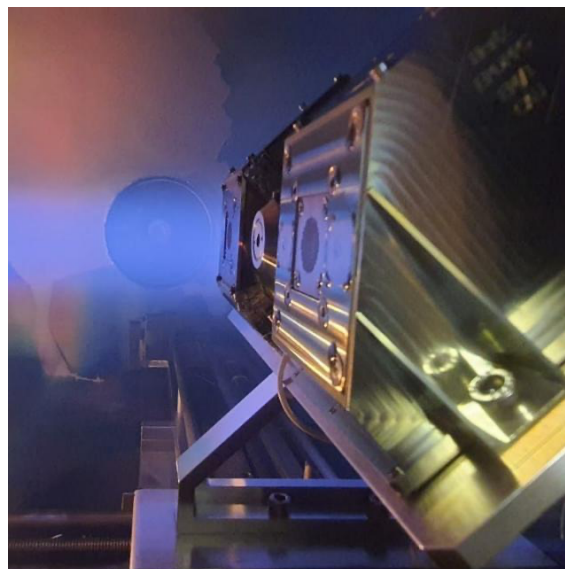
<sup>3</sup>von Hoerner & Sulger GmbH, Schwetzingen, Germany

## Abstract:

Since the first demonstration of an electrically propelled spacecraft with an ion engine different ion sources have been realized. They replaced the traditional chemical thrusts on satellites for e.g. attitude control or geosynchronizing the orbit. In the first place, thrust measurements are the most direct approach for the characterization of a thruster for spacecraft propulsion. Sophisticated instruments, like direct and indirect thrust balances, have been developed, which are able to quantify the thrust with accuracies of a few  $\mu\text{N}$  [1]. Other diagnostics are based either on electric methods, like faraday cups and retarding potential analysers [2,3], or on optical methods, like emission spectroscopy. Recently, force probes for momentum flux measurements are complementing these on-ground instruments [4]. The non-electrostatic techniques are important because it is well-known that the exhaust plumes of plasma-based thrusters contain a significant amount of energetic neutral particles, which can not be detected with electrostatic diagnostics. Therefore, thrust predictions based on electrostatic techniques may significantly underestimate the performance of a thruster.

Additionally, electric thrusters, in particular plasma-based ion thrusters, are known for generating secondary plasma that interacts with the spacecraft. These spacecrafts often carry sensitive instruments, solar panels and other subsystems that might be disturbed or degraded by the unintended backflow from the applied thruster. In test facilities, it is very difficult to numerically calculate or mimic these very small unintended side effects. For this purpose, special plasma diagnostics, i.e., the Electric Propulsion Diagnostic Package (EPDP), have been developed to analyse the plasma surrounding an electrically driven spacecraft [5].

**Keywords:** plasma diagnostics, electric spacecraft propulsion, ion thrusters, sputtering, charge-exchange collisions



**Figure 1:** Measurements with two sensors and a Faraday cup in a test chamber with a broad beam ion source.

## References:

1. Polk, J.E. et al., 33rd International Electric Propulsion Conference, IEPC-2013-440 (2013)
2. Mazouffre, S. et al., 35th International Electric Propulsion Conference, IEPC-2017-336 (2017)
3. Trottenberg, T. et al., 36th International Electric Propulsion Conference, IEPC-2019-345 (2019)
4. Trottenberg, T. et al., EPJ Techn. Instrum. 5, 3 (2018)
5. Trottenberg, T. et al., EPJ Techn. Instrum. 8, 16 (2021)



# Experimental Observation of plasma parameter and photoresist ashing in an inductively coupled plasma sources with MRWPT antenna

Ju-Ho Kim <sup>1,\*</sup>, Young-Hun Hong <sup>1</sup>, Chin-Wook Chung <sup>1</sup>

<sup>1</sup> Department of Electrical Engineering, Hanyang University, Seoul, Republic of Korea

\* electricrckjh@gmail.com

## Abstract:

Recently, several studies have reported that the efficient plasma generation and uniformity control are possible in an inductively coupled plasma with a passive resonant antenna for magnetic resonance wireless power transmission (MRWPT antenna). In this study, the plasma parameter and photoresist ashing are observed in an inductively coupled plasma source with MRWPT antenna. A powered antenna connected to RF generator through a matching network and a MRWPT antenna are installed above a quartz plate on the top of the chamber. As the self-resonance frequency of MRWPT antenna is adjusted, the plasma distribution is measured at both plasma bulk and wafer-level. The results are compared with the PR ashing results.

**Keywords:** ICP source, wireless power transfer, magnetic resonance, Photoresist asing, plasma density distribution control.

## References:

1. M. A. Lieberman and A. J. Lichtenberg (2005) Principles of plasma discharges and materials processing, John Wiley & Sons
2. H. J. Lee, H. C. Lee, Y. C. Kim and C. W. Chung (2013) Control of plasma density distribution via wireless power transfer in an inductively coupled plasma, *Plasma Sources Sci. Technol.* **22** 032002
3. J. H. Kim, Y. H. Hong and C. W. Chung (2019) High efficient plasma generation in an inductively coupled plasma using a passive resonant antenna, *Plasma Sources Sci. Technol.* **28** 105018
4. T. W. Kim, M. Y. Lee, Y. H. Hong, M. H. Lee, J. H. Kim, and C. W. Chung (2021) Local electron and ion density control using passive resonant coils in inductively coupled plasma, *Plasma Sources Sci. Technol.* **30** 025002

## Experimental observation of hysteresis in a neon inductively coupled plasma at low pressure

Young-Hun Hong <sup>1,a</sup>, Tae-Woo Kim <sup>1</sup>, Moo-Young Lee <sup>2</sup>, Ju-Ho Kim <sup>1</sup>, and Chin-Wook Chung <sup>1,\*</sup>

<sup>1</sup> Department of Electrical Engineering, Hanyang University, 17 Haengdang-dong, Seongdong-gu, Seoul 133-791, Republic of Korea

<sup>2</sup> Department of Nanoscale Semiconductor Engineering, Hanyang University, 17 Haengdang-dong, Seongdong-gu, Seoul 133-791, Republic of Korea

<sup>a</sup> hyh4736@gmail.com, <sup>\*</sup> joykang@hanyang.ac.kr

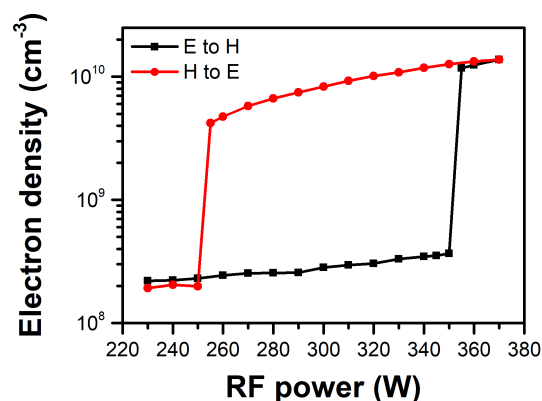
### Abstract:

A hysteresis of an electron density and a coil current is observed in an inductively coupled plasma at low gas pressure. Generally, the hysteresis has been reported at high pressure Ramsauer gas discharges, but in the neon plasmas, the hysteresis is observed at low pressure (5 mTorr). Electron energy distribution functions (EEDFs) are measured with increasing and decreasing RF power to analyze this hysteresis phenomenon. The measured EEDF has a Maxwellian distribution in the E mode discharge while it has a bi-Maxwellian like distribution in the H mode discharge. This implies that the electrons of the high energy are efficiently heated in the H mode discharge and therefore the collisional energy loss is possibly reduced.

**Keywords:** E to H transition, Neon inductively coupled plasma, Hysteresis, EEDF, Heating mechanism

### References:

1. M. A. Lieberman and A. J. Lichtenberg (2005) Principles of plasma discharges and materials processing, John Wiley & Sons
2. H. C. Lee and C. W. Chung (2015) Effect of Electron Energy Distribution on the Hysteresis of Plasma Discharge: Theory, Experiment and Modeling, *Sci. Rep.*, 5 15254



**Figure 1:** Measured electron density versus the RF power in neon ICP at 5 mTorr.

# Spatial and Temporal Characterization of NO and OH Concentration in a Nanosecond Pulsed Surface Dielectric Barrier Discharge Plasma Using Picosecond Laser Induced Fluorescence

L. Ibba<sup>1</sup>, P. F. Ambrico<sup>2</sup>, F. Avino<sup>1</sup>, I. Furno<sup>1</sup>

<sup>1</sup> Swiss Plasma Center, Ecole Polytechnique Fédérale de Lausanne (EPFL), Lausanne, Switzerland

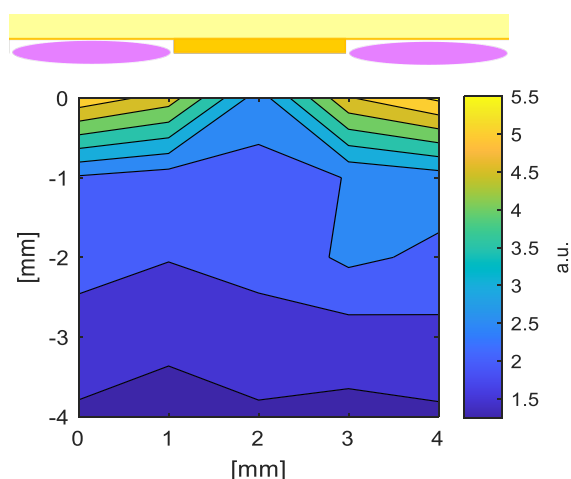
<sup>2</sup> Institute for Plasma Science and Technology – CNR, Bari, Italy

## Abstract:

The mechanisms involved in plasma sterilization are still unclear. The reactive oxygen and nitrogen species (RONS) produced by the plasma seem to have a dominant role in the processes involved in bacteria inactivation. Hence, their complete characterization is necessary to further our understanding of the complex interaction between plasmas and bacteria. Here, we focus on NO and OH, being two key molecules in the chemistry of atmospheric pressure, low temperature plasmas generated in air. Besides having anti-microbial potential and being directly involved in the bacteria inactivation processes, they are also precursor of other relevant molecules produced by the plasma like  $N_xO_y$ ,  $HNO_3$ ,  $H_2O_2$ ,  $O_3$  [1][2]. We perform NO and OH density measurements through Laser Induced Fluorescence (LIF) at atmospheric pressure using a diode-pumped high energy picosecond Nd:YAG laser to excite the molecules and an Intensified Charge Coupled Device (ICCD) to detect the fluorescence light. This allows resolving the decay of the fluorescence light and correct the fluorescence signal intensity according to the collisional quenching of the background gas, mainly caused by  $H_2O$ . Absolute calibration is performed using a gas mixture with a known amount of NO concentration. A Surface Dielectric Barrier Discharge (SDBD) is powered by a nanosecond pulse high voltage power supply to generate a 400 nanoseconds pulse plasma discharge at atmospheric pressure in a controlled synthetic air flow (80%  $N_2$  / 20%  $O_2$ ) with variable humidity content. The NO concentration is measured in a 2-dimensional area 4x4 mm in front of the plasma surface at different relative humidities. The results show the NO concentration to drastically drop a few millimeters from the plasma surface, suggesting that the NO survives only in the proximity of the plasma, where is produced, quickly reacting with other molecules. Between two consequent plasma discharges at 1 kHz the NO concentration does not vary significantly, but it increases with larger humidity content, sug-

gesting  $H_2O$  improves NO production. LIF measurements of OH are currently undergoing and will also be discussed.

**Keywords:** low temperature plasma, atmospheric pressure, picosecond laser induced fluorescence, surface dielectric barrier discharge, SDBD, nanosecond pulse plasma, collisional quenching, nitric oxide, NO, hydroxyl radical, OH, humidity



**Figure 1:** NO concentration in arbitrary units in a 4x4 mm square perpendicular to the plasma surface. Above the plot is a schematic of the position of the plasma (pink) and the electrode (orange). The measurements were taken at  $2.8 \pm 3\%$  RH, 500  $\mu s$  after the plasma discharge.

## References:

1. X. L. Hao, A. M. Mattson, C. M. Edelblute, M. A. Malik, L. C. Heller, and J. F. Kolb, Nitric Oxide Generation with an Air Operated Non-Thermal Plasma Jet and Associated Microbial Inactivation Mechanisms *Plasma Process. Polym.* 11(11), 1044–1056 (2014).
2. D. Xu, D. Liu, B. Wang, C. Chen, Z. Chen, D. Li, Y. Yang, H. Chen, M.G. Kong, In situ OH generation from  $O_2^-$  and  $H_2O_2$  plays a critical role in plasma-induced cell death, *PLoS ONE*, 10 (6) (2015), Article e0128205

# Dielectric barrier discharge plasma reduction of iron oxides

V. Udachin<sup>1</sup>, S. Dahle<sup>1,2</sup>, W. Maus-Friedrichs<sup>1</sup>

<sup>1</sup> Clausthal University of Technology, Clausthal-Zellerfeld, Germany

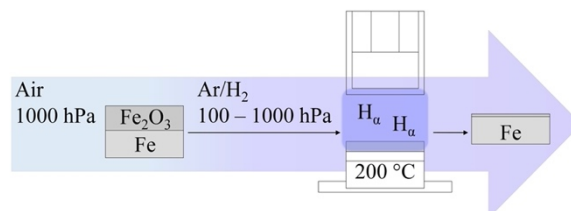
<sup>2</sup> University of Ljubljana, Ljubljana, Slovenia

## Abstract:

Native surface oxides, which usually contaminate metals in ambient atmosphere, limit their performance in industrial applications like coating or welding. For the reason of removal of such oxide layers, different deoxidation techniques were developed. Up to date, non-thermal hydrogen plasmas have been shown as efficient in deoxidation of metals. Whereas low pressure plasma deoxidation techniques have been studied intensively, there is only a minimal information about deoxidation effect of hydrogen plasmas, which operate in the atmospheric pressure range. Promising results have been obtained for dielectric barrier discharge (DBD) hydrogen plasmas in deoxidation of copper oxides at room temperature and 100 – 1000 hPa [1,2]. Nevertheless, no information about deoxidation effect of a DBD plasma on other metals, which are widely used in fabrication processes, is available.

In the study, we report the application of a DBD plasma for deoxidation of natively oxidized iron surfaces in an Ar/H<sub>2</sub> atmosphere at 100 and 1000 hPa (Figure 1). Plasma treatments were performed at a discharge voltage of 10 kV and a frequency of 8.8 kHz at room temperature as well as in the combination with heating. The chemical state and the morphology of the samples before and after treatments were studied via X-ray photoelectron spectroscopy (XPS) and atomic force microscopy (AFM), respectively. The results revealed, that iron surface lattice oxide was reduced to clean metal after several seconds of plasma treatment at the elevated temperatures in the range from 200 to 300 °C, whereas room plasma treatment did not lead to an efficient deoxidation. Additionally, a plasma was characterized using optical emission spectroscopy (OES).

**Keywords:** dbd plasma, deoxidation, iron deoxidation, surface treatment, hydrogen plasma, cold plasma, industrial applications



**Figure 1:** Illustration of the experimental process of deoxidation of natively oxidized iron surfaces in an Ar/H<sub>2</sub> DBD plasma as well as the results of deoxidation

## Acknowledgements:

The project was funded by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) – Project-ID 394563137 – SFB1368.

## References:

1. Udachin, V., Wegewitz, L., Dahle, S., Maus-Friedrichs, W. (2022). Reduction of copper surface oxide using a sub-atmospheric dielectric barrier discharge plasma. *Applied Surface Science*, 573, 151568.
2. Xu, Z. J., Qi, B., Di, L. B. (2013). On the Mechanism of Copper Oxide Reduction by Dielectric Barrier Discharge Plasma Using H<sub>2</sub> and Ar Mixture Gases. *Advanced Materials Research*, 690, pp. 1664-1667. Trans Tech Publications Ltd.

# Deposition in silicon trenches and porous substrate using Bipolar High power impulse magnetron sputtering

S. Atmane<sup>1</sup>, N. Rochdi<sup>1</sup>, A. Caillard<sup>1</sup>

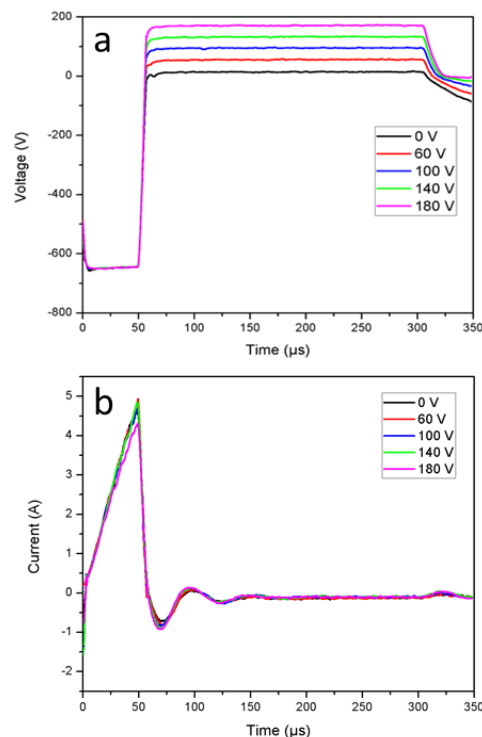
<sup>1</sup> GREMI, Université d'Orléans, CNRS, 14 rue d'Issoudun, BP6744, 45067 Orléans cedex 2, France

## Abstract:

High power impulse magnetron sputtering (HiPIMS) is a physical deposition method of thin films in the vapor phase. This technique provides larger plasma densities and ion fluxes to a substrate compared to the direct current magnetron sputtering (DCMS). HiPIMS discharges operate in pulsed mode with short widths and low duty cycle. As a consequence, ion densities are 2-3 orders of magnitude higher compared to DCMS [1-2]. Bipolar high power impulse magnetron sputtering (Bp-HiPIMS) refers to the application of a positive voltage pulse following the conventional negative HiPIMS pulse. Recently this method has attracted a great interest for the deposition of thin and dense layers due to the ability to control the ion energy and the flux. The effects of a positive pulse following the negative HiPIMS pulse have been intensively studied using energy-resolved mass spectrometry whereas few papers deal with the effect of this pulse on the properties of hard coatings. This includes exploring the influence of varying the length of the positive voltage pulse ( $U_{rev} = 10-200$  V) following a typical HiPIMS pulse on the ion-energy distribution function (IEDF) of the various ions [3].

In this study, we investigated the effect of the positive pulse on the filling of silicon trenches and on the penetration inside porous substrate (silicon), the filling and the penetration being characterized by scanning electron microscopy and Rutherford backscattering spectroscopy. We correlated these results to the time integrated (and time resolved) ion energy distribution obtained by an energy-resolved mass spectrometry and by a retarding field energy analyzer, to the energy influx incoming onto the substrate measured by a thermal probe.

**Keywords:** Bipolar HiPIMS, Sputtering, Plasma.



**Figure 1:** (a) Discharge voltage  $U_{ref}(t)$  and (b) current  $I_D(t)$  waveforms recorded during standard hipims and bipolar HiPIMS with a reverse 250  $\mu s$  long positive voltage pulse of (0-180V) applied immediately following the negative pulse.

## References:

1. Hecimovic, A., Burcalova, K., Ehiasarian, A.P. (2008) Origins of ion energy distribution function (IEDF) in high power impulse magnetron sputtering (HIPIMS) plasma discharge, *J. Phys. D: Appl. Phys.* 41, 095203
2. Jablonka, L., Moskovkin, P., Zhang, Z., Zhang, S.-L., Lucas, S., Kubart, T. (2019) Metal filling by high power impulse magnetron sputtering, *J. Phys. D: Appl. Phys.* 52365202.
3. Keraudy, J., Viloan, R.P.B., Raadu, M.A., Brening, N., Lundin, D., Helmersson, U. (2019) *Surf. Coat. Technol.* 359433–7.

# Plasma / liquid interactions during low pressure plasma sputtering deposition of Pt nanoparticles on liquid glycerol

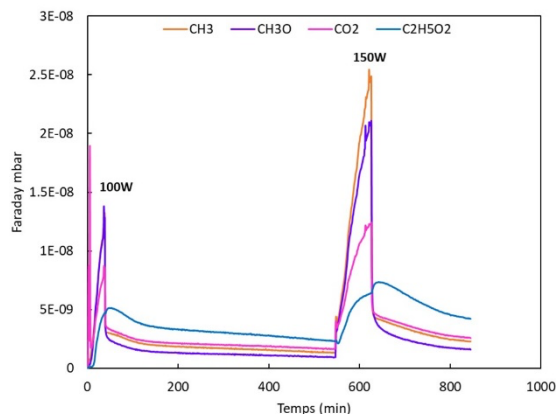
S. Atmane<sup>1</sup>, P. Brault<sup>1</sup>, A. Caillard<sup>1</sup>

<sup>1</sup> GREMI, Université d'Orléans, CNRS, 14 rue d'Issoudun, BP6744, 45067 Orléans cedex 2, France

## Abstract:

Pt nanoparticles (NPs) are widely used as catalysts for oxygen reduction reactions (ORR) in electrochemical systems as the Proton Exchange Membrane fuel cell (PEMFC). NPs can be obtained through various physical, chemical or physicochemical routes. The chemical methods are very versatile in terms of controlling NPs shape and size but requires additives, which generate by-products difficult to remove and NPs with limited purity. In contrast, physical methods as magnetron sputtering on solid substrates avoid the use of additives, allowing the production of pure metallic NPs. In order to make the magnetron sputtering process compatible with conventional liquid ink preparation techniques used for manufacturing fuel cell, we recently reported the synthesis of Pt NPs over a host liquid substrate (glycerol) that sustains low pressures [1]. Molecular dynamic simulations highlighted that the NPs diffusion in the liquid phase depends on the associated kinetic energy of Pt atoms when arriving on the liquid surface and so the plasma / liquid interaction properties. In this study, we investigated these interactions and the gas phase properties using experimental techniques as (an energy resolved) mass spectrometer and energy flux probe giving us access to the energy distribution of the sputtered species, total energy influx incoming onto the liquid and the gas phase composition (see Figure 1). These results are correlated to the NPs physical properties obtained by X Ray Diffraction/Diffusion and High Resolution Transmission Electron, enabling us a better understanding of the Pt NPs growth phenomena on and in the liquid phase.

**Keywords:** plasma sputtering, nanoparticles, plasma – liquid interactions.



**Figure 1:** Time evolution of the gas phase composition obtained using a mass spectrometer (HPR Hiden analytical).

## References:

1. Orozco-Montes, V. Caillard, A., Brault, P., Chamorro-coral, W., Bigarre J., Sauldubois, A., Andreazza, P., Cuynet, S., Baranton, S., Coutanceau, C (2021), Synthesis of Platinum Nanoparticles by Plasma Sputtering onto Glycerol: Effect of Argon Pressure on Their Physicochemical Properties, *J. Phys. Chem. C*, 125, 5, 3169–3179.



# Kinetic Monte-Carlo modeling of SiN:H thin film deposition by PECVD on complex substrates: characterization of air-connected porosity and improvement by ion beam assisted deposition

J. Müller<sup>1,\*</sup>, P. Moskovkin<sup>1</sup>, S. Lucas<sup>1,2</sup>

<sup>1</sup> Laboratoire d'Analyse par Réactions Nucléaires (LARN), Namur Institute of Structured Matter (NISM), University of Namur, Namur, Belgium

<sup>2</sup> ICS (Innovative Coating Solutions)

## Abstract:

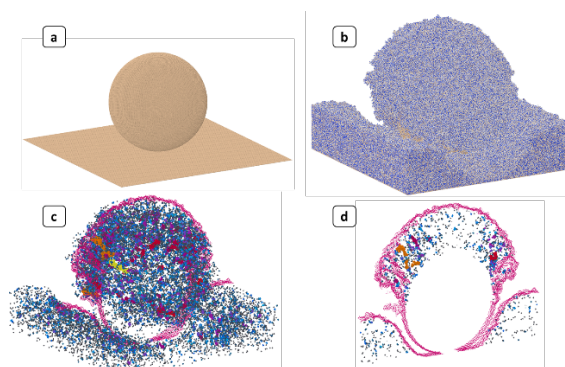
Thin film technology presents a large range of applications in various fields like anti-scratching coatings, optoelectronic devices, optical coatings... However, physical behavior of such thin films highly depends on intrinsic properties (film structuration, porosity, elasticity,...) and it is mandatory to develop efficient analytical or numerical methods to predict such properties.

In the case of devices sensible to oxidation and/or moisture like OLED (organic light emitting diode), thin film encapsulation is required and it is critical to detect air-connected pores to avoid the deterioration of such device.

The purpose of this work is to simulate the SiN:H encapsulation of complex objects by PECVD using SiH<sub>4</sub> as a precursor, in order to predict pores, especially those connected to the surface. To do so, we will first deposit in-silico a 3D SiN:H coating on complex shaped substrate by means of kinetic Monte-Carlo code NASCAM [1,2]. Special care is put on defining the chemical reactions occurring in the gas phase [3] and at the coating surface [4]. Then, the porosity is computed to detect critical zones where the substrate can be exposed to moisture and oxidation. Figure 1 shows the modeling of a SiN:H porous coating deposited on a 2  $\mu$ m diameter spherical substrate and the pores detected inside the coating. The pink dots represent the air-connected surface. We can notice that the bottom of the object is not coated, and then not protected. The sphere has been selected to take into account in our modelling curved and lateral sides and possible shadowing which are representative of any configuration that can be found in OLED coating.

Finally, we investigate different methods (rotating or conformal source, ion beam assisted deposition) allowing to improve the density of the coating and to reduce the presence of air-connected pores.

**Keywords:** OLED, encapsulation, PECVD, ion beam assisted deposition, kinetic Monte-Carlo, atomistic scale modelling.



**Figure 1:** Figure illustrating the different steps of modelling. (a) 2  $\mu$ m diameter sphere in silicon to be coated. (b) Sphere coated by 1  $\mu$ m thick SiN:H film deposited by PECVD. (c) Pores detection in the coating. (d) Vertical cross-section of the 3D pore map: the pink dots represent the air-connected surface.

## References:

1. R. Tonneau et al. (2021) "Understanding the role of energetic particles during the growth of TiO<sub>2</sub> thin films by reactive magnetron sputtering through multi-scale Monte Carlo simulations and experimental deposition". *J. Phys. D. Appl. Phys.* 2.
2. <https://www.incosol4u.com/nascam-general>
3. K. Goran et al. (2017) "Reactions in silicon-nitrogen plasma", *Phys. Chem. Chem. Phys.*
4. H. Caquineau et al. (1997) "Influence of the reactor design in the case of silicon nitride PECVD", *Chemical Engineering Science*

## The influence of the RF- power on the photocatalytic properties of thin PE-ALD ZnO films deposited at room temperature

Daria Jardas<sup>1,2,\*</sup>, Robert Peter<sup>1,2</sup>, Iztok Turel<sup>3</sup>, Mladen Petravić<sup>1,2</sup>, Aleš Omerzu<sup>1,2</sup>

<sup>1</sup> Department of Physics, University of Rijeka, Rijeka, Croatia

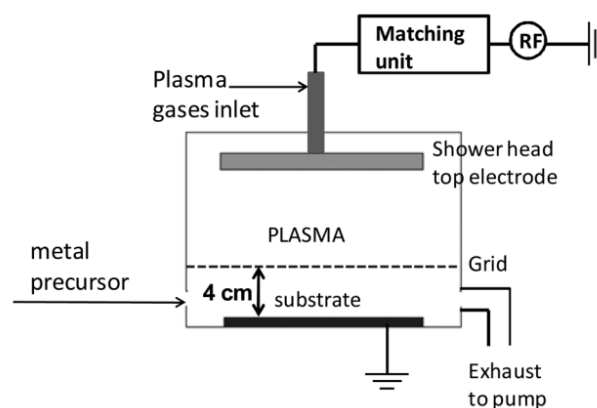
<sup>2</sup> Centre for Micro- and Nanosciences and Technologies, University of Rijeka, Rijeka, Croatia

<sup>3</sup> Faculty of Chemistry and Chemical Technology, University of Ljubljana, Ljubljana, Slovenia

### Abstract:

Atomic layer deposition (ALD) is a sophisticated deposition technique that can be used to synthesize high-quality thin ZnO films. Furthermore, films with different structural and physical properties can be obtained by varying the synthesis parameters [1,2,3,4]. The most important parameter is the deposition temperature - it determines the structure and material properties of the deposited films [2,3]. The range of conventional (i.e. thermal) ALD deposition temperatures for high-quality ZnO is between 120 °C and 200 °C, and the best photocatalytic activity was observed for films deposited above 150 °C [2,3]. This characteristic makes this technique unsuitable for deposition on organic and biological substrates. However, a variant of the ALD technique which involves plasma - plasma-enhanced atomic layer deposition (PE – ALD) allows deposition at lower temperatures without compromising film quality and growth rate and even improving some of the films properties. For example, the films deposited at 80 °C using the PE-ALD method have been shown to have ten times better photocatalytic efficiency than films deposited at the same temperature using ALD [1]. Moreover, the PE-ALD technique allows synthesis at room temperature, which makes it suitable for temperature-sensitive substrates. In addition to temperature, plasma RF power is also an important parameter in PE-ALD synthesis [3,4]. In the present study, we compare the optical properties of thin films deposited by the PE - ALD method at room temperature and power between 50 W and 300 W. For this purpose, UV-Vis spectroscopy and photoluminescence measurements were undertaken.

**Keywords:** zinc oxide, thin film, atomic layer deposition, plasma, photocatalysis



**Figure 1:** PE-ALD configuration of the Beneq TFS 200 ALD reactor used for synthesis [5].

### References:

1. A. Omerzu et al. (2021), *Surfaces and Interfaces*, 23, 100984.
2. R. Peter et al. (2020), *J. Phys. Chem. C*, 124 (16), 8861-8868.
3. Pilz et al. (2018) *J. Vac. Sci. Technol. A*, 36, 01A109
4. M. Napari et al. (2017) *Surf. Coat. Technol.* 326, 281-290
5. M. Kariniemi et al. (2011), *Chem. Mater.* 23, 2901

# Influence of adatoms on the titanium nano-cone formation during helium ion bombardment

F. Sanchez <sup>1,\*</sup>, L. Marot <sup>1</sup>, R. Antunes <sup>1</sup>, R. Steiner <sup>1</sup>, J. Spicher <sup>1</sup>, P. Lattner <sup>1</sup>, M. Kisiel <sup>1</sup>, D. Mathys <sup>3</sup>, E. Meyer <sup>1</sup>, M. Astasov-Frauehoffer <sup>2</sup>, I. Hauser-Gerspach <sup>2</sup>, S. Kühl <sup>4</sup>, J. Köser <sup>5</sup>, R. Wagner <sup>6</sup>, J. Hofstetter <sup>6</sup>, K. Mukaddam <sup>4</sup>

<sup>1</sup> Department of Physics, University of Basel, Klingelbergstrasse 82, 4056 Basel, Switzerland

<sup>2</sup> University Center for Dental Medicine Basel (UZB), University of Basel, Mattenstrasse 40, 4058 Basel, Switzerland

<sup>3</sup> Swiss nanoscience Institute, University of Basel, Klingelbergstrasse 50/70, CH-4056 Basel, Switzerland

<sup>4</sup> Department of Oral Surgery, University Center for Dental Medicine Basel (UZB), University of Basel, Mattenstrasse 40, 4058 Basel, Switzerland

<sup>5</sup> Institute for Chemistry and Bioanalytics, School of Life Sciences, FHNW, Hofackerstrasse 30, 4132 Muttensz, Switzerland

<sup>6</sup> Institut Straumann AG, Peter Merian-Weg 12, 4052 Basel, Switzerland

## Abstract:

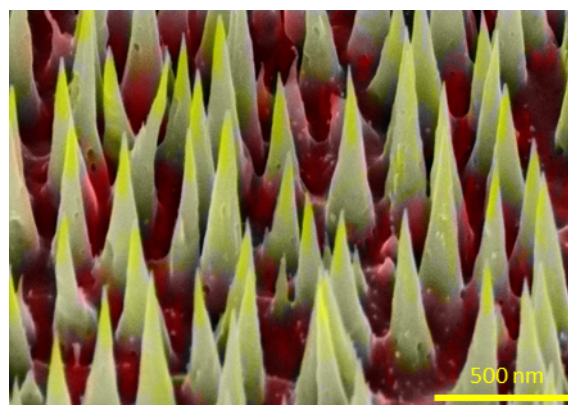
Firstly discovered on cicada wings, the surface patterning with spikes shows interesting antibacterial properties. By reproducing a similar structure on titanium, preliminary work has showed the possibility of developing antibacterial properties [1]. In a cooperative project bringing together physicists, biologists, dentists and dental implant producers we aim at developing dental implants with such antibacterial properties using helium ions to produce spikes on a titanium surface.

For this study, an unbalanced magnetron sputtering (UBM) or a Kaufmann ion source (eH1000) were used to provide He ions onto 15 mm titanium discs. Metallic contaminants (Ti, Fe or/and W) were deposited during irradiation. The He ion flux ( $2\text{--}10 \times 10^{19} \text{ m}^{-2}\text{s}^{-1}$ ) and energy were determined by the use of homemade faraday cups and a retarding field energy analyzer (Semion single sensor by Impedans).

Structured samples were produced at set temperatures of 613 K and characterized with scanning electron microscopy (SEM), focused ion beam (FIB). Metallic contaminants were quantified by means of quartz microbalance (QMB) and/or X-ray photoelectron spectroscopy (XPS). This work aims at studying topology modifications due to the presence of contaminants in the ion beam. At fixed fluences, the growth rate of the spikes were enhanced by a factor about 10 when compared to pure He ion bombardment. The mechanisms involved in the cone formation will be discussed in this contribution.

## Keywords:

Titanium surface structuration, metallic contamination, He ions, scanning electron microscopy, focused ion beam, quartz microbalance, X-ray photoelectron spectroscopy



**Figure 1:** SEM micrograph of titanium surface after exposure to He ions forming spikes of about 600 nm height.

## References:

1. K. Mukaddam et al., Antibacterial and Cell-Adhesive Effects of Bio-Inspired Nanostructured Materials, European Cells and Materials. <http://ecmconferences.org/abstracts/2017/Collection3/collection3.pdf>.

# Properties of (W,Zr)B<sub>2-z</sub> Protective Coatings Deposited by RF Magnetron Sputtering Method

T. Mościcki \*, R. Psiuk, J. Chrzanowska-Giżyńska

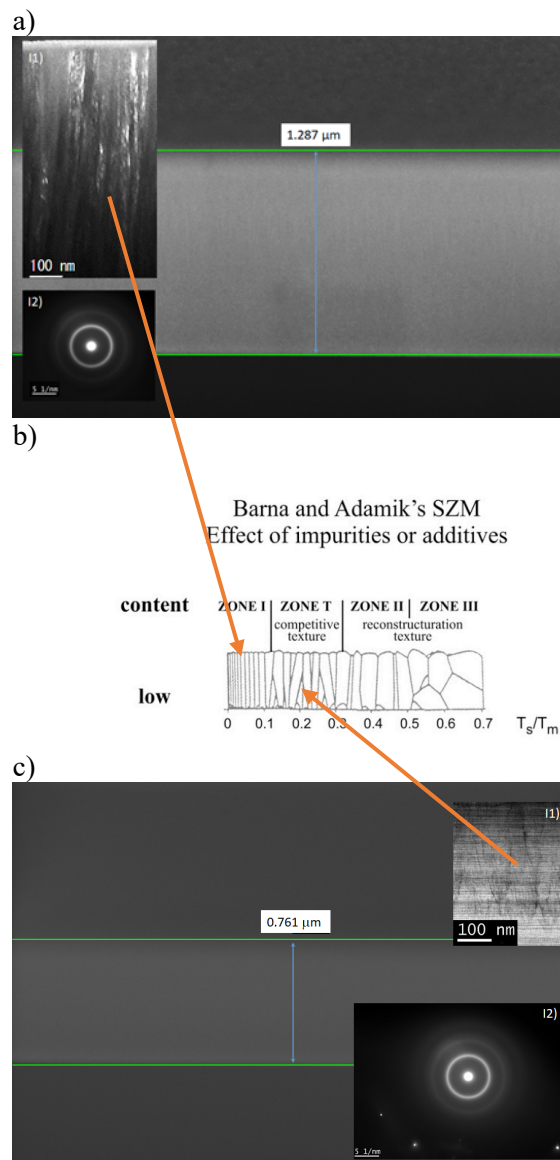
<sup>1</sup> Institute of Fundamental Technological Research PAS, Warsaw, Poland

## Abstract:

Plasma-deposited protective and tribological coatings have a significant role in many areas of today's industry. In tools, industry coatings lead to increased tool life and a larger variety of work-pieces from different materials. The most popular thin films based on nitrides lead to a significant improvement in the life of tools and the quality of machined parts [1]. However, there are applications in which nitride coatings are not enough to fulfil the requirements. In this work magnetron sputtered WB<sub>2</sub> coatings doped with 8, 11 and 16 at% zirconium were analysed using energy dispersive spectroscopy, X-ray diffraction and nanoindentation tester. Deposited coatings are superhard and having hardness above 44.5 GPa. W-Zr-B coatings have a hardness higher than WB<sub>2-z</sub> and at the same time have a lower Young modulus due to change of microstructure (Fig. 1). Thermal treating tests (annealing and cycling thermal loads) indicate higher stability of Zr doped coatings. The annealed coatings possess better mechanical properties because they are less prone to cracking and have a lower Young's modulus. In the case of cycling thermal loads the hardness and Young's modulus of coatings grow due to thermal residual stresses. Also changing of unstable  $\alpha$ -WB<sub>2</sub> to  $\omega$ -W<sub>2</sub>B<sub>5</sub> phase is observed in the case of coating without zirconium. Plasma - deposited W-Zr-B films with very high hardness, improved resistivity to cracking and good thermal stability are competitive to commonly used nitrides and are a good candidate as a protective and tribological coatings.

**Keywords:** protective coatings, magnetron sputtering, tungsten borides, superhard materials, thermal stability

**Acknowledgement** This work was funded by project SUPERCOAT; project number: TECHMASTRATEG-III/0017/2019



**Figure 1:** . Cross-section of deposited layers. a) SEM , BF STEM (insert 1) images and SAED pattern (insert 2) of W-B coating, b) effect of the amount of added element into one-phase coating on its microstructure according to Barna and Adamik structural zone model (SZM) c) W-Zr-B coating, 16 at% zirconium

## References:

1. Haubner, R., Lessiak, M., Pitonak, R., Köpf, A., Weissenbacher, R. (2017) Evolution of conventional hard coatings for its use on cutting tools, *Int. J. Refract. Met. H.*, 62, 210-218.



# Adsorption of Organic Components from Fluid Mixtures on Cold Plasma Coated Aerogels in Supercritical Fluid Chromatography: Experiment and Simulation

I. Jung<sup>1</sup>, B. Schroeter<sup>1</sup>, P. Gurikov<sup>2</sup>, I. Smirnova<sup>1</sup>

<sup>1</sup> Institute for Thermal Separation Processes, Hamburg University of Technology, Hamburg, Germany

<sup>2</sup> Laboratory for Development and Modelling of Novel Nanoporous Materials, Hamburg University of Technology, Hamburg, Germany

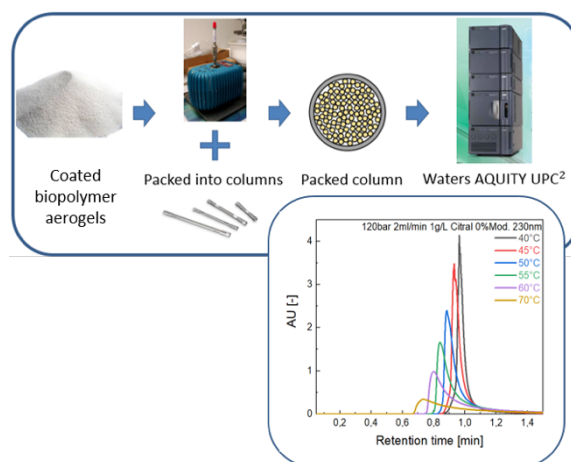
## Abstract:

Aerogels are mesoporous materials characterized by special properties: high pore volumes ( $> 90\%$  v/v), high specific surfaces (up to  $1200 \text{ m}^2/\text{g}$ ) and low densities ( $< 0.2 \text{ g/cm}^3$ ). In presented research, biopolymer aerogel microparticles (e.g., derived from alginate, chitosan, cellulose and proteins) are produced via supercritical  $\text{CO}_2$ -drying and used as stationary phase in supercritical fluid chromatography (SFC). The physico-chemical interactions with various solutes are determined. In particular, adsorption behavior of various organic solutes in dependence of the microstructural and surface properties are investigated. Since functionalization and change of aerogels exterior and inner surface energy can be achieved via cold plasma treatment, pristine as well as plasma-coated aerogels are compared.

Aerogel particles (diameter  $< 50 \mu\text{m}$ ) were prepared by emulsion gelation or milling processes. Cold plasma coating of the aerogels with  $\text{C}_4\text{F}_8$  was performed subsequently to applicate nanometer-precise hydrophobic and superhydrophobic polymer layers. Commercial stainless-steel columns were then filled with the biopolymer aerogel particles serving as stationary phase. Columns with non-plasma treated aerogels were used as reference. Measurements were performed using an Acquity UPC<sup>2</sup> SFC (Waters, Milford, USA); the fluid phase consisted of pure  $\text{CO}_2$  and an aromatic organic solute (e.g. caffeine). Interactions between the solutes and modified biopolymer aerogels measured as retention data were determined with quantitative models. A modified van't-Hoff equation was applied to calculate the temperature dependent adsorption enthalpy of different solutes from the mobile phase to the stationary phase. Overall, the use of supercritical fluid chromatography for characterization of adsorption behavior with substrates is demonstrated, covering a wide range of different pore structures and surface energies. As next step, a cellular automaton model will be formulated to describe the diffusion dynamics and adsorption

behavior of solutes on modified biopolymer surfaces in supercritical phases.

**Keywords:** aerogels, cold plasma coating, mesoporous structure, supercritical fluid chromatography, biopolymers



**Figure 1:** Method development: determination of the adsorption behaviour of org. substances in the SFC using coated aerogels particles.

## Acknowledgment:

The funding of the presented research in the framework of research training group GRK 2462 by German Research Foundation (Deutsche Forschungsgemeinschaft, DFG) is gratefully acknowledged

## References:

1. Schroeter, B.; Jung, I., Bauer, K.; Gurikov P.; Smirnova, I. (2021) Hydrophobic Modification of Biopolymer Aerogels by Cold Plasma Coating, *Polymers.*, 13, 3000.
2. Sun, M.; Ruiz Barbero, S.; Johannsen, M.; Smirnova, I.; Gurikov, P. (2019) Retention characteristics of silica materials in carbon dioxide/methanol mixtures studied by inverse supercritical fluid chromatography, *J. Chromatogr. A.*, Vol. 15888.

## Photoreactive pressure sensitive adhesives - bacterial and fungal properties

K. Mozelewska<sup>1\*</sup>, P. Niezgoda<sup>2</sup>

<sup>1</sup> West Pomeranian University of Technology in Szczecin, Faculty of Chemical Technology and Engineering, Department of Organic Chemical Technology and Polymer Materials, Szczecin, Poland

<sup>2</sup> West Pomeranian University of Technology in Szczecin, Faculty of Environmental Management and Agriculture, Department of Environmental Management and Agriculture, Szczecin, Poland

### Abstract:

According to the definition of PSTC (Pressure-Sensitive Tapes Council), pressure-sensitive adhesives in a dry state (after separation of the solvent) show aggressive and permanent stickiness. A characteristic feature of this group is also strong adhesion to the surface, which occurs without the need to apply a force greater than finger pressure [1]. Recently, more and more attention has been paid to natural sources of substances with antibacterial and antioxidant properties. Essential oils are used in various industries due to their numerous properties. Essential oils are volatile, natural mixtures of compounds characterized by intense fragrances. They consist of a mixture of several to several hundred compounds, mainly hydrocarbons (terpenes and sesquiterpenes) and oxidized compounds (aldehydes, ketones, alcohols, acids, phenols, esters, and others) [2].

In the first stage of work, polyacrylate pressure-sensitive adhesives based on acrylate copolymers were obtained. For their preparation, commercially available reagents of technical purity were used: butyl acrylate, acrylic acid, 2-ethylhexyl acrylate, and methyl acrylate. Moreover, in the presented work, 4-acryloyloxybenzophenone (ABP) was used. On the other hand, the role of the radical initiator was played by AIBN. The next stage was the modification of the obtained adhesive with the use of various essential oils. Then, the antimicrobial properties of the modified adhesives were determined by the smear method. Material tested: Gram-negative bacteria - *Escherichia coli* - strain NCTC 12241 / ATCC 25922, Gram-positive bacteria - *Staphylococcus epidermidis* - strain NCTC 13360 / ATCC 12228, and *Candida albicans* fungi - strain NCPF 3255 / ATCC 2091. Frozen *Escherichia coli* and *Staphylococcus epidermidis* bacteria were thawed at room temperature, inoculated into the appropriate broth and incubated 48 h at 37 °C. Thereafter, 300 µl of the bacterial suspension was spread with a sponge on each of the culture plates with the appropriate medium. In the case of *Candida albicans* fungi, they were thawed at room temperature and inoculated onto cultivated for 5 days at

25 °C. The smear method was used - the mycelium for plating on the plates was prepared by adding to 8 ml of NaCl - water for the dilution of the cultivated mycelium, and then vortexed. The obtained suspension was inoculated on the culture plates with SDA medium with the use of a plate spreader. Cultured for 5 days at 25 °C. Trials with the tested material were carried out: 3 discs soaked with the tested adhesive were placed on a given plate. The bacteria plates were incubated for 48 h at 37 °C, while the *Candida albicans* plates were incubated for 5 days at 25 °C. After this time, the results were read.

**Keywords:** pressure-sensitive adhesives, self-adhesives tape, bacterial and fungal properties

### References:

1. Pandey; V., Fleury, A., Villey, R., Creton, C., Ciccotti, M. (2020) Linking peel and tack performances of pressure sensitive adhesives. *Soft Matter*, 16, 3267-3275.
2. Sangermano, M. (2012), Advances in cationic photopolymerization., *Pure Appl. Chem.*, 84, 2089-2101



# Decontamination of *Drosophila suzukii* Infested Blueberries with Low-pressure Cold Plasma Treatment

P. Starič<sup>1\*</sup>, N. Cvelbar Weber<sup>2</sup>, M. Resnik<sup>1</sup>, K. Vogel Mikuš<sup>1,3</sup>, I. Junkar<sup>1</sup>

<sup>1</sup>Department of Surface Engineering, Jožef Stefan Institute, Ljubljana, Slovenia

<sup>2</sup>Department of Fruit Growing, Viticulture and Oenology, Agricultural Institute of Slovenia, Ljubljana, Slovenia

<sup>3</sup>Biotechnical Faculty, University of Ljubljana, Ljubljana, Slovenia

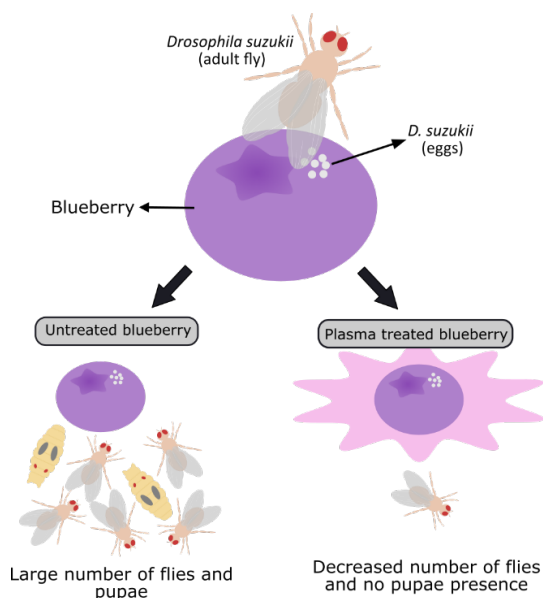
## Abstract:

Blueberries (*Vaccinium spp.*) are an important food source of vitamins, anthocyanins, and antioxidants. The global consumption demand is rising and it is foreseen the same trend in the following years. However, fruit post-harvest life is relatively short and is subjected to decay and mechanical damage. Other organisms, such as fruit flies also cause fruit damage.

*Drosophila suzukii*, so called the spotted wing drosophila, originates from Asia. However, in Europe and America, it is becoming a severe pest concern of soft fruits such as strawberries, raspberries, and blueberries. The *D. suzukii* lays its eggs in fresh and healthy fruits, and its developing larva then damages the fruits directly. The wounds from the oviposition of eggs can also cause the colonization of secondary pathogens, which indirectly causes the damage of fruits.

Cold plasma technology is slowly gaining its way to the field of agriculture. It has potential use in the decontamination technologies of food, fruits, and seeds. Our research addressed the increasing problem of *D. suzukii* infections of blueberries. The fruits were infected with *D. suzukii* and later exposed to different cold plasma treatment conditions. The X-ray photoelectron spectroscopy (XPS) confirmed chemical changes on the blueberry surface after plasma treatment. After plasma treatment of berries, the samples were incubated and examined for the presence and number of adult fruit flies and pupae. The preliminary results show that treatment of blueberries with oxygen plasma could be used for the successful decrease in number of *D. suzukii* adult flies and pupae formation in blueberry fruits.

**Keywords:** *Drosophila suzukii*, spotted wing drosophila, pupae cold plasma, blueberries, decontamination, XPS, chemical changes



**Figure 1:** Schematic representation of the research, where *Drosophila suzukii* infected blueberries were exposed to cold plasma discharge. Plasma-treated samples exhibited a decrease in the number of adult fruit flies after the incubation period compared to untreated samples.

## References:

1. Entling, W., Anslinger, S., Jarausch, B., Michl, G., Hoffmann, C. (2019). Berry skin resistance explains oviposition preferences of *Drosophila suzukii* at the level of grape cultivars and single berries. *Journal of Pest Science*, 92(2), 477-484.
2. Dong, X. Y., Yang, Y. L. (2019). A novel approach to enhance blueberry quality during storage using cold plasma at atmospheric air pressure. *Food and Bioprocess Technology*, 12(8), 1409-1421.

# Deposition of microencapsulated essential oil extracted from Neem seeds on the surface of plasma pretreated cellulose knitwear

T. Perinović<sup>1</sup>, A. Ludaš<sup>1\*</sup>, S. Ercegović Ražić<sup>1</sup>

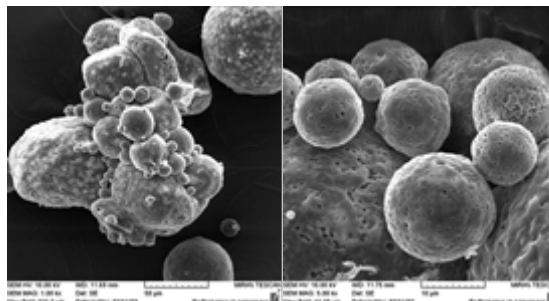
<sup>1</sup> Department of Materials, Fibres and Textile Testing, University of Zagreb Faculty of Textile Technology, Zagreb, Croatia

## Abstract:

In this paper, microencapsulation of Neem essential oil (*Azadirachta indica*) was carried out by solvent evaporation technique. Cellulose knitwear was treated with Neem essential oil, which has a wide range of antibacterial properties. Before treatment, cellulose knitwears were pretreated with oxygen and argon low-pressure cold plasma under defined conditions. The synthesized microcapsules were applied to the textile material by a spraying method. Surface analysis of treated samples were done by FE-SEM microscope, while the microbiological tests by Agar diffusion.

Microencapsulation has proven to be a successful, environmentally friendly and economically viable technology for commercial application in the pharmaceutical, agrochemical industries, and more recently in the textile industry [1]. It represents the most effective way to store a substance, as it allows controlled release of the active ingredient. Essential oils are evaporative, natural and complex ingredients characterized by a strong odor. They originate from aromatic plants as secondary metabolites [2]. Essential oil extracted from Neem seeds has a broad spectrum of antibacterial activity against gram-negative and gram-positive microorganisms, and also shows an inhibitory effect on the growth of various pathogens, including bacteria, fungi and viruses [3]. Due to the increasing environmental demands imposed by modern textile technological processes, great importance is attached to environmentally friendly treatments. Special interest in the application of plasma technology in the textiles material science has been noted in the last two decades, especially in the processes of the textile surface modification and finishing, to obtain multifunctional textile products [4].

**Keywords:** Neem essential oil, microencapsulation, plasma pretreatment, textile materials.



**Figure 1:** FE-SEM images of synthesized microcapsules (1000 and 5000 x magnifications)

## References:

1. Brlek, I.; Ludaš, A.; Sutlović, A. (2021), Synthesis and Spectrophotometric Analysis of Microcapsules Containing Immortelle Essential Oil, *Molecules*, Vol. 26 (8), pp. 1-9
2. Boh, B.; Knez, E. (2006), Microencapsulation of essential oils and phase change materials for applications in textile products, *Indian journal of fibre & textile research*, Vol. 31, pp. 72-82
3. Eid, A.; Jaradat, N.; Elmarzugi, N. (2017), A Review of chemical constituents and traditional usage of Neem plant (*Azadirachta indica*), *Palestinian Medical and Pharmaceutical Journal*, Vol. 2 (2), pp. 75-81
4. Ercegović Ražić, S.; Čunko, R. (2009), Property modifications of textiles using plasma, *Tekstil* Vol. 58 (3), pp. 55-74.

## Acknowledgement:

This work has been supported by European Union, through European Social Fund under the project UP.03.1.1.04.0024 „Development and implementation of professional practice at Faculty of Textile Technology“. The content of the paper is the exclusive responsibility of University of Zagreb Faculty of textile technology.



# Implementation of a Non-Thermal Atmospheric Pressure Plasma for Eradication of Plant Pathogens from a Surface of Economically Important Seeds

Jakub Orłowski<sup>1</sup>, Agata Motyka-Pomagruk<sup>1</sup>, Anna Dzimitrowicz<sup>2</sup>, Weronika Babinska<sup>1</sup>, Dominik Terefinko<sup>2</sup>, Michał Rychlowski<sup>3</sup>, Michał Prusinski<sup>1</sup>, Paweł Pohl<sup>2</sup>, Ewa Lojkowska<sup>1</sup>, Piotr Jamroz<sup>2</sup>, Wojciech Sledz<sup>1</sup>

<sup>1</sup> Laboratory of Plant Protection and Biotechnology, Intercollegiate Faculty of Biotechnology University of Gdansk and Medical University of Gdansk,

University of Gdansk, 58 Abrahama, 80-307 Gdansk, Poland;

<sup>2</sup> Department of Analytical Chemistry and Chemical Metallurgy, Wrocław University of Science and Technology, 27 Wybrzeże St. Wyspiańskiego, 50-370 Wrocław, Poland;

<sup>3</sup> Laboratory of Virus Molecular Biology, Intercollegiate Faculty of Biotechnology University of Gdansk and Medical University of Gdansk, University of Gdansk, 58 Abrahama, 80-307 Gdansk, Poland;

## Abstract:

Plant diseases cause huge losses in the agricultural sector every year [1]. Fighting phytopathogens with the use of pesticides is not ideal due limited number of targeted microorganisms and toxicity. Application of cold atmospheric pressure plasmas (CAPPs) might be a solution to this need. Antibacterial and plant growth stimulating action of CAPPs results from the generated UV radiation in addition to reactive oxygen and nitrogen species.

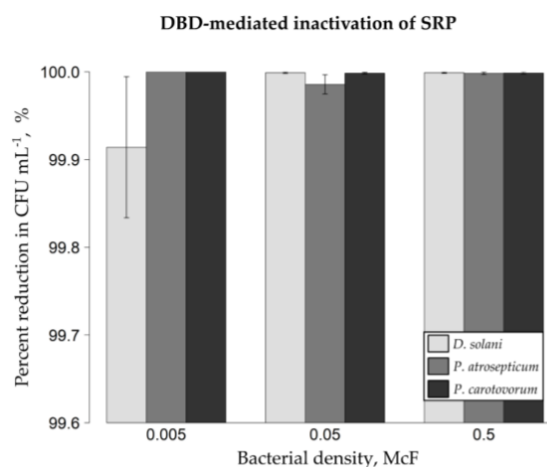
The aim of this study was to examine antibacterial properties of a direct action of a dielectric barrier discharge (DBD) plasma against *Dickeya* and *Pectobacterium* spp. inoculated on glass sphere models or mung bean seeds. Besides, the influence of DBD plasma action on the germination and growth of mung bean seeds was investigated. Helium was used as discharge gas. The DBD exposure lasted from 1 to 5 min, depending on the conducted experiment.

In terms of eradication of phytopathogens from glass spheres, the noted reductions in CFU ml<sup>-1</sup> exceeded 99.93% (3.16 log) (Figure 1). Concerning elimination of bacterial cells from mung bean seeds, reductions over 99.91% (3.07 log) were observed. Due to the application of optical emission spectrometry (OES), transmission electron microscopy (TEM), and confocal laser scanning microscopy (CLSM), we found that the generated reactive oxygen and nitrogen species (RONS) probably led to the denaturation and aggregation of DNA, proteins, and ribosomes. The cellular membrane disrupted, leading to an outflow of the cytoplasm from the DBD-exposed cells. 2 min DBD plasma treatment stimulated by 3-4% seed germination rate and by up to 13.4%

subsequent early growth of seedlings. Negative effects of 4 min DBD action were noted shortly after the treatment, however these effects were no longer observable or reduced to 9.7% after the extended incubation periods.

It was proven that the direct DBD plasma treatment can be an efficient tool to fight bacterial phytopathogens without impeding plant growth. Broad application of this technology into agriculture can be foreseen due to its eco-friendliness and antimicrobial potency. As artificial plasmas can be generated with the use of various types of electric discharges, further modifications and optimization of this method would allow for scaling-up this technology. [2]

**Keywords:** cold atmospheric pressure plasma, CAPP, dielectric barrier discharge plasma, DBD, non-thermal plasma; *Pectobacteriaceae*; soft rot erwinias; plant protection; agriculture; *Vigna radiate*; mung beans



**Figure 1:** Inactivation efficacy of soft rot *Pectobacteriaceae* from mung bean seeds with the use

of dielectric barrier discharge as the nonthermal atmospheric pressure plasma source. Time of the exposure of inoculated seeds to DBD plasma was 2 min. Percentage reductions in the amount of colony-forming units (CFU) per milliliter of Ringer buffer were shown. Either 0.5, 0.05 or 0.005 McF bacterial suspension had been used for inoculating disinfected mung bean seeds. Means  $\pm$  standard errors are depicted. *D. solani* IFB0099, *P. atrosepticum* IFB5103, and *P. carotovorum* IFB5118 strains were used. DBD—dielectric barrier discharge; McF—McFarland scale; SRP—Soft rot *Pectobacteriaceae*) [2]

#### References:

1. Borkar, S.G.; Yumlembam, R.A. Bacterial Diseases of Crop Plants; Taylor and Francis Group: London, UK, 2017
2. Motyka-Pomagruk, A.; Dzimitrowicz, A.; Orlowski, J.; Babinska, W.; Terefinko, D.; Rychlowski, M.; Prusinski, M.; Pohl, P.; Lojkowska, E.; Jamroz, P.; et al. Implementation of a Non-Thermal Atmospheric Pressure Plasma for Eradication of Plant Pathogens from a Surface of Economically Important Seeds. *Int. J. Mol. Sci.* 2021, 22, 9256.

# Slip Resistance Improvement for Shoe Outsoles through Atmospheric-Pressure Plasma Treatments

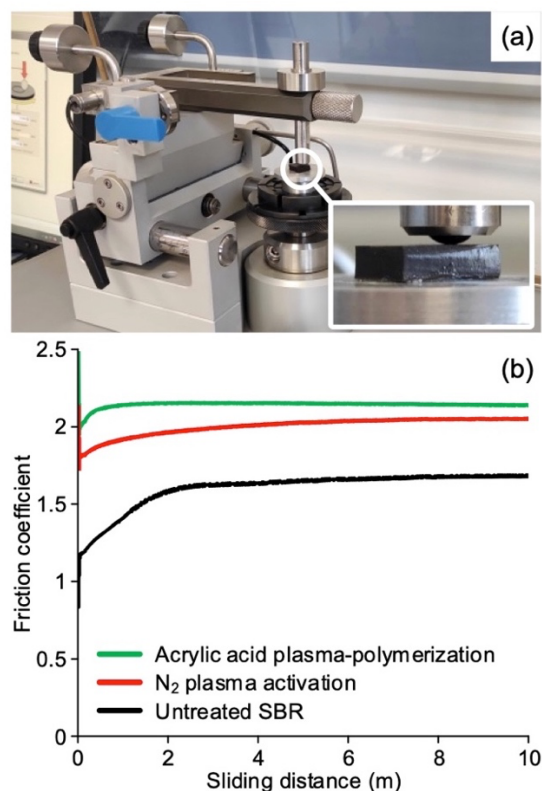
R. Múgica-Vidal <sup>1,\*</sup>, I. Muro-Fraguas <sup>1</sup>, A. Sainz-García <sup>1</sup>, E. Sainz-García <sup>1</sup>, A. González-Marcos <sup>1</sup>, F. Alba-Elías <sup>1</sup>

<sup>1</sup> Department of Mechanical Engineering, University of La Rioja, Logroño, Spain

## Abstract:

Accidental slips and falls are among the main causes of injury and death in work places.<sup>[1]</sup> Therefore, the slip resistance of shoe outsoles is fundamental to prevent these accidents and preserve the safety and health of the user. In our previous work, surface activation and plasma-polymerization of coatings by an atmospheric-pressure plasma jet (APPJ) system were proved successful for promoting the adhesive properties of outsole materials.<sup>[2,3]</sup> Thus, the bonding strength between the vulcanized rubber of the outsole and the leather of the upper part of the shoe was improved. In the current study, activation treatments with air and N<sub>2</sub> plasma jets using different combinations of process parameters, as well as coatings of different plasma-polymerized liquid precursors, were used for improving the slip resistance of styrene-butadiene rubber (SBR), which is a commonly used material for manufacturing outsoles. The effects of the treatments in the sliding resistance of the SBR were evaluated by measuring the friction coefficient of untreated and treated SBR samples in tribological tests using a steel ball as the sliding counterpart (Figure 1a). The higher the friction coefficient, the greater the slip resistance. The best of the activation treatments, which used N<sub>2</sub> to generate the plasma jet, achieved an increase of 24.6% in the friction coefficient of the SBR. On the other hand, an increase of 33.3% in the friction coefficient of the SBR was achieved by a coating of plasma-polymerized acrylic acid, which gave the best results in this study (Figure 1b). According to the results of the tribological tests and physico-chemical characterizations, the studied treatments could improve the slip resistance of the SBR in two ways: (1) roughening the rubber surface through an etching effect of the plasma, and (2) incorporating polar groups to the surface chemistry.

**Keywords:** slip resistance, friction coefficient, tribology, atmospheric-pressure plasma jet, activation, plasma-polymerization, footwear



**Figure 1:** (a) Tribological test setup and close view of the contact between the steel ball and an SBR sample, (b) friction coefficient graphs of untreated SBR and samples with the best activation and plasma-polymerization treatments.

## References:

1. Iraqi, A., Vidic, N. S., Redfern, M. S., Beschorner, K. E. (2019), Prediction of coefficient of friction based on footwear outsole features, *Appl. Ergon.*, 82, 102963.
2. Múgica-Vidal, R., Mercadal-Guillén, J., Alba-Elías, F., Sainz-García, E. (2021) Improvement of the adhesive capacity of SBR for footwear outsoles by surface activation and coating deposition with atmospheric pressure plasma, *Plasma Process. Polym.*, 18, 2100046.
3. Múgica-Vidal, R., Mercadal-Guillén, J., Sainz-García, E., Alba-Elías, F. (2021) Atmospheric-pressure plasma treatments of NBR for the improvement of adhesion in footwear applications, *Int. J. Adhesion Adhes.*, 108, 102865



# Tribological Performance of 2D Nano-coated Steel Surfaces

M.J.G. Guimarey<sup>1,2,\*</sup>, M. Hadfield<sup>1</sup>, A. Abdelkader<sup>1</sup>

<sup>1</sup> Department of Design and Engineering, Bournemouth University, Talbot Campus, Poole, United Kingdom

<sup>2</sup> Nafomat Group, Department of Applied Physics, iMATUS, University of Santiago de Compostela, Santiago de Compostela, Spain

## Abstract:

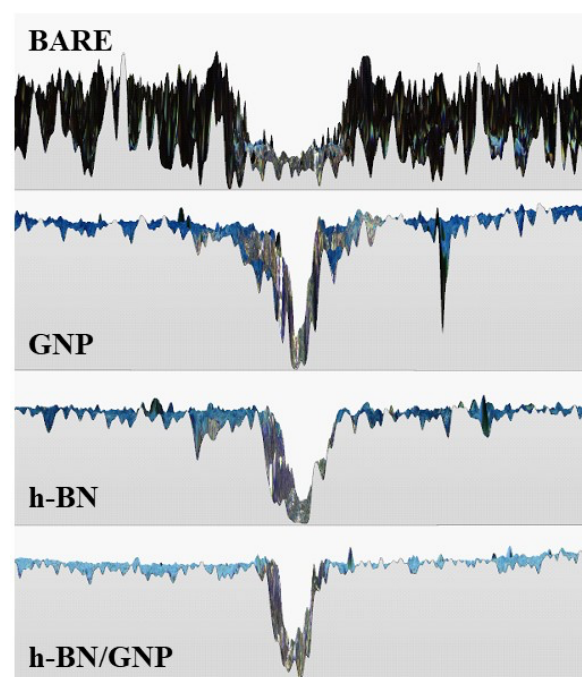
Reducing friction losses and thus wear between moving mechanical parts in contact with each other remains one of the significant industrial challenges of recent years [1]. The synergy between nanomaterials and industrial lubricant has shown great promise in addressing this challenge with reported excellent friction and wear performance [2]. However, liquid lubricants may not be used under certain operating conditions or applications (e.g. high temperature or vacuum). Therefore, the solid self-lubrication of two-dimensional (2D) nano-additives as metal reinforcement coatings presents itself as a very interesting alternative [3]. Moreover, nano-coatings would be a solution to the current demand for energy efficiency and help reduce the production of waste oil.

Among the 2D nanomaterials, graphene (GNP) and hexagonal boron nitride (h-BN) stand out as protective coatings due to their layered structure and excellent chemical resistance, barrier properties, impermeability together with thermal stability. Thus, to explore their potential in improving steel's friction and wear resistance, films of GNP, h-BN and a heterogeneous structure of both (h-BN/GNP) were deposited on mild steel by spray coating method combined with post-heat treatment. The thickness and composition of the coatings were analysed using scanning and transmission electron microscopy (SEM and TEM). The friction pure sliding tests to evaluate the tribological capability of the coatings compared with bare steel were performed using a ball-on-disc tribometer under dry and lubricated conditions. The wear produced on the coated substrates was analysed by 3D optical profilometer. In friction tests with sliding steel-steel tribo-pairs under dry conditions, graphene-containing decreased the coefficient of friction and wear area by 21% and 31%, respectively. The h-BN-graphene hetero-structure exhibits more anti-wear properties than stand-alone 2D materials (Figure 1). This research suggests that this sort of nano-coating has the potential to improve the tribological performance of steel-steel tribo-pairs.

Furthermore, this study has revealed the outstanding friction and wear resistance of these

nano-coatings and the possibility of nano-coatings with an extraordinary anti-corrosion oxidation resistance potential.

**Keywords:** Graphene Nanoplatelets; Hexagonal Boron Nitride; Coating; Friction; Wear.



**Figure 1:** Wear cross-section profiles produced on the steel substrates after the lubricated friction sliding tests for the uncoated and coated samples.

## References:

1. Holmberg, K., Kivikytö-Reponen, P., Härkisaari, P., Valtonen, K., Erdemir, A. (2017) Global energy consumption due to friction and wear in the mining industry, *Tribol. Int.* 115 116-139.
2. Guimarey, M.J.G., Abdelkader, A., Comuñas, M.J.P., Alvarez-Lorenzo, C., Thomas B., Fernandez, J., Hadfield, M. (2020) Comparison between thermophysical and tribological properties of two engine lubricant additives: electrochemically exfoliated graphene and molybdenum disulfide nanoplatelets, *Nanotechnology*, 32, 025701-025714
3. Scharf, T.W., Prasad, S.V. (2013) Solid lubricants: a review. *J. Mater. Sci.* 48, 511–531.

# Effect of electrochemistry on tribochemical wear of monocrystalline silicon

Chen Xiao<sup>1,2</sup>, Feng-Chun Hsia<sup>1,2</sup>, Bart Weber<sup>1,2</sup> and Steve E. Franklin<sup>1,3,\*</sup>

<sup>1</sup>Advanced Research Center for Nanolithography (ARCNL), Science Park 106, 1098XG, Amsterdam, Netherlands

<sup>2</sup>Van der Waals-Zeeman Institute, IoP, University of Amsterdam, Science Park 904, 1098XH, Amsterdam, Netherlands

<sup>3</sup>Department of Materials Science and Engineering, The University of Sheffield, Sheffield S1 3JD, UK

\*Corresponding author: s.franklin@arcnl.nl (S.E. Franklin).

## Abstract:

Tribochemical wear that causes atomic attrition before material yield has attracted lots of attention due to its critical role in ultra-precision manufacturing, micro-electromechanical system (MEMS) and reliable microscopy.<sup>1</sup> As a chemical means or processes initiated from contact interface, the potentiality of tribochemical reactions must be determined by the states of treated surface and the external energy (such as mechanical energy and electric energy).<sup>1</sup> Further understanding of regulating tribochemical reactions could provide insights into reducing friction and wear (in MEMS applications) or improving machining efficiency (in nanomanufacturing).

By using an atomic force microscope with an electrochemical cell (EC-AFM, Bruker, Icon), the tribochemical removal between SiO<sub>2</sub> microsphere and silicon surface was conducted in electrolyte solution (3% wt.% sodium chloride solution), as shown in Figure 1. During the tests, the displacement amplitude was set as 500 nm, the sliding velocity was 2 μm/s, and the applied voltage was constant at 1.5 V.

Different from mechanical removal processes involving abrasion, fracture, or plastic deformation, the tribochemical wear relies on the shear-induced hydrolysis reaction of Si substrate with existing water molecules in the sliding environment. Quantified wear results on silicon surface indicates that tribochemical removal rate follows the Arrhenius kinetics law depended on the stress-assisted chemical reactions which is described as a thermally activated atom-by-atom removal process with the lower energy barrier reduced by external mechanical energy. Tribochemical wear ( $\sim 4 \times 10^4$  kcal/mol) consumes only 5.6% dissipated energy of mechanical wear case ( $\sim 7 \times 10^5$  kcal/mol), which is attributed to the reduced energy barrier of Si-Si bond breakage and no lattice distortion underneath the machined area. And this value is further reduced by introducing electrochemistry. Figure 2 shows the simplified energy coordinate along the dissociation of Si-Si

bond at silicon substrate. Tribo/Electrochemical reactions induced by applied mechanical/electrical energy promote the wear rate by reducing the energy barrier:  $E_a = E_{\text{Thermal}} - E_{\text{Tribochemistry}} - E_{\text{Electrochemistry}}$ , through lowering the activated energy and increasing the initial energy, respectively.

**Keywords:** Material removal; Silicon; Tribochemistry; Electrochemistry;

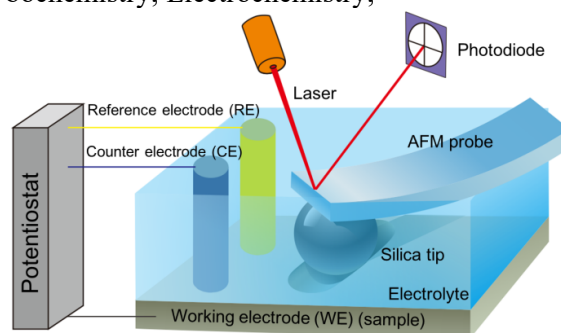


Figure 1: Schematic illustration of the tribochemical removal on silicon surface against a silica microsphere ( $R \sim 1 \mu\text{m}$ ) attached to an AFM cantilever in EC-AFM system.

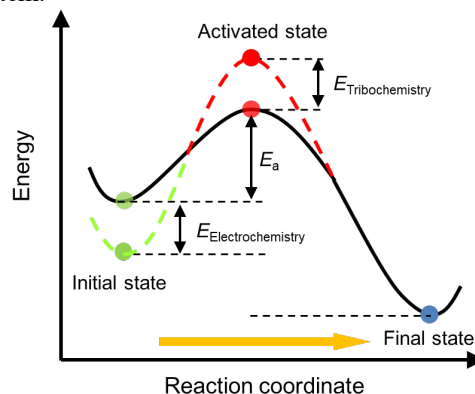


Figure 2: Schematic energy diagram along the reaction coordinate leading to the dissociation of Si-Si bond at silicon surface with the assistant of tribochemical and electrochemical reactions.

## References:

1. Chen, L. et al., "Nanomanufacturing of silicon surface with a single atomic layer precision via tribochemical reactions" Nat. Commun, 9, 2018, 1542.

# Tribological and corrosion properties of coating systems dried by infrared radiation

I. Stojanović<sup>1</sup>, L. Turkalj<sup>1\*</sup>, I. Cindrić<sup>1</sup>, I. Juraga<sup>1</sup>, D. Rakela Ristevski<sup>2</sup>

<sup>1</sup>Faculty of Mechanical Engineering and Naval Architecture, University of Zagreb, Croatia

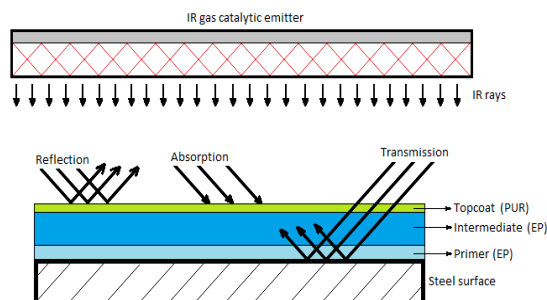
<sup>2</sup>Končar Steel Structures Inc., Zagreb, Croatia

## Abstract:

In many fields of industry, abrasion and erosion processes are dominant wear mechanisms that reduce lifetime of machine parts. Because of that, abrasion and erosion resistance must be taken into consideration due to coating wear over time which leads to premature corrosion and material breakdown. Coatings based on epoxy primers provide superior mechanical properties, such as hardness, durability, resistance to impacts, wear, and chemicals while maintaining exceptional adhesiveness, thus providing great protection in different corrosive environments. However, epoxy layers exposed to sun are prone to chalking due to the action of ultraviolet (UV) rays dissolving the binder. Because of that, polyurethane (PUR) topcoats are applied due to their high chemical durability, low vapour permeability, resistance to solutions, protecting epoxy layers from UV rays with appealing gloss and appearance. Due to high abrasion, impact, and UV resistance, the defined coating system has proven to be an excellent corrosion and wear protection solution for top decks, exposed areas of car parks, exterior ship surfaces, sun decks, helipads, balconies, and offshore platforms. Also, coatings must have a low environmental impact. More and more drastic environmental regulations aiming to reduce the emission of volatile organic compounds (VOC) in the atmosphere have led to developing water-based solvent coatings which are increasingly used. In this paper, water-borne and solvent-borne coating systems, with and without zinc in the primer, dried by infrared (IR) radiation (Figure 1) and under atmospheric conditions were studied. To assess these differences, abrasion, erosion, hardness, and adhesion tests of the coating were performed. Anti-corrosion performance of the coatings was characterised using a salt spray chamber. Zinc rich (Zn-R) coatings showed better tribological properties, with IR-dried coating systems displaying higher abrasion resistances. Solvent-borne coatings exhibited better corrosion properties than water-borne coatings when exposed to accelerated corrosion test in salt spray chamber. Both IR and air-dry coating curing technologies achieved adequate results with the

difference of IR significantly reducing drying time.

**Keywords:** abrasion, erosion, coating hardness, coating resistance, IR drying



**Figure 1:** IR curing process of 3-layer coating system.

## References:

1. Liu D., Zhao W, Liu S., Cen Q, Xue Q (2015) Comparative tribological and corrosion resistance properties of epoxy composite coatings reinforced with functionalized fullerene C60 and graphene, *Surf. Coat. Technol.*, 286, 354-364.
2. Li J., Chen P., Wang Y. (2020) Tribological and corrosion performance of epoxy resin composite coatings reinforced with graphene oxide and fly ash cenospheres, *J. Appl. Polym. Sci.*, 138.

# Nonlinear Dynamic Analysis of Slot-Type Externally Pressurized Air Bearing System

C.C. Wang

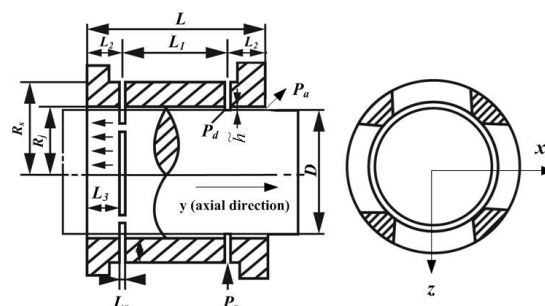
Graduate Institute of Precision Manufacturing, National Chin-Yi University of Technology, Taichung, Taiwan

## Abstract:

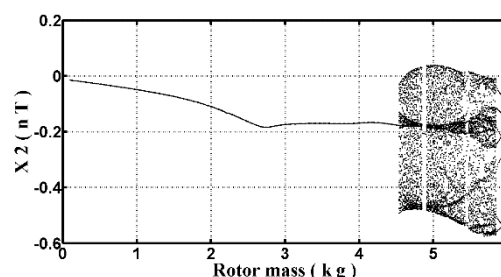
Slot-type externally pressurized air bearing (SEPAB, Figure 1) systems have been extensively used for a variety of mechanical engineering application, and potential for use in high-rotational speed, high stiffness, and high-precision instrumentation. SEPABs have two major advantages including the air supply externally and slot restrictor design, and also provide multi-directional supporting forces and higher stiffness to increase the greater rotational stability. However, under certain operating conditions, SEPAB systems exhibit non-periodic or chaotic motion as the result of a nonlinear pressure distribution within the gas film, gas supplied imbalances, an inappropriate design, and so forth. So, in order to understand and control when the bearing system occurs non-periodic motions and under what kind of operating conditions, the dynamic response of the SEPAB system are analyzed by using different numerical methods, namely a perturbation method and a hybrid numerical scheme combining the finite difference method and the differential transformation method. The key performances of SEPAB obtained by these two methods are compared and verified. The dynamic behavior of rotor center in SEPAB are examined under different operating conditions by bifurcation diagram (Figure 2), Poincaré maps, power spectra and maximum Lyapunov exponents (MLE) etc. Changes in the operated parameters produce various vibration behaviors in the horizontal and vertical directions of rotor, including periodic, subharmonic, quasi-periodic and chaotic motions. In particular, when the rotor mass is high, the bearing system exhibits unstable nonlinear chaotic behavior. The major contribution of this study is its finding that the rotor mass and bearing number influence the stability of SEPAB system. Furthermore, this study confirmed that the rotor exhibits chaotic behavior for specific ranges of the rotor mass and bearing number. The MLE is a key index for defining stable and unstable regions (nonchaotic and chaotic regions, respectively) for the SEPAB system over various values of the rotor mass and bearing number. The research results can be used as an analysis reference and important basis for avoiding unstable

nonlinear behaviors in SEPAB system design and application.

**Keywords:** slot-type externally pressurized, air bearing, chaotic motion, bifurcation diagram, Poincaré maps, power spectra, maximum Lyapunov exponents



**Figure 1:** Structure of slot-type externally pressurized air bearing.



**Figure 2:** Bifurcation diagrams of the rotor center in the SEPAB system for different masses and a bearing number of 3.0.

## References:

1. Ni, G., Chen, J., Wang, H. (2019) Degradation assessment of rolling bearing towards safety based on random matrix single ring machine learning, *Saf Sci.*, 118, 403-408.
2. Wang, C.C., Lee, R.M., Yau, H.T., Lee, T.E. (2019) Nonlinear analysis and simulation of active hybrid aerodynamic and aerostatic bearing system, *J Low Freq Noise Vibr Act Control.*, 38(3-4), 1404-1421.



## Characterization and simulation of tribological behaviour for WC spherical surfaces with AlTiSiN coating

M.Marqués<sup>1</sup>, A.Claver<sup>1</sup>, J.A.García<sup>1,2</sup>, D. Salcedo<sup>1</sup>, A. Tellez<sup>1</sup>, I. Quintana<sup>3</sup>

<sup>1</sup>Materials science, Public University of Navarre, Pamplona, Spain

<sup>2</sup>INAMAT (Institute for Research in Advanced Materials), Pamplona, Spain

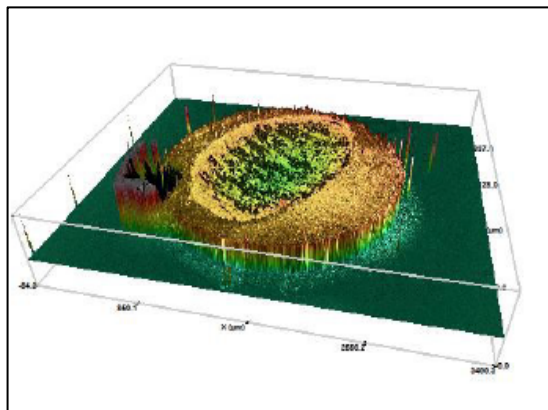
<sup>3</sup>Tekniker Research & Technology Centre, Eibar, Spain

### Abstract:

Nowadays there is a greater demand for treatments and coatings that improve performance and increase the useful life of work tools used in various industrial applications. Through this study it is intended to replicate the machining of difficult-to-machine materials such as titanium or stainless steels using coated WC balls that simulate the work of tools. This work analyzes the improvement of the tribological properties of AlTiSiN coating deposited by PVD on spherical tungsten carbide surfaces and adds a finite element simulation to confirm the wear theory. Friction and wear tests were carried out by using a ball-on-disk tribometer and an optical profilometer (Figure 1). Scanning electron microscope technique (SEM) were employed to measure the stoichiometry and thickness of the different films. The products of wear and tear generated by the interaction of the test materials were measured by EDS. The results point that AlTiSiN coating increase significantly the wear resistance versus different materials like 316L stainless steel and Titanium.

### Keywords:

PVD, Hard coatings, Tungsten carbide, Titanium, Micro hardness, Wear, Friction, Tribology, Simulation, Finite elements.



**Figure 1:** Figure illustrating the wear of a spherical surface carried out by a pin on disc experiment. The ball is coated by a WC layer and the plate used in front is raw titanium. The rendering has been made with a confocal microscope by SENSOFAR S.L.

### References:

1. R. J. Rodríguez, J. A. García, A. Medrano, M. Rico, R. Sánchez, R. Martínez, C. Labrugère, M. Lahaye and A. Guette, "Tribological behaviour of hard coatings deposited by arc-evaporation PVD", Vacuum, Vol. 67, pp. 559-566, 2002.

# Optimized surface engineering solutions for glass mould industry

J. Costa<sup>1</sup>, J.P. Dias<sup>1</sup>, A. Cavaleiro<sup>1,2</sup>

<sup>1</sup>LED&Mat-IPN, Instituto Pedro Nunes, Coimbra, Portugal

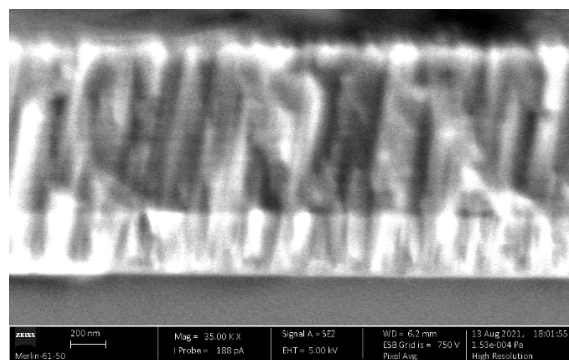
<sup>2</sup>SEG-CEMMPRE, Departamento de Engenharia Mecânica - Universidade de Coimbra, Coimbra, Portugal  
jcosta@ipn.pt

## Abstract:

The pressure imposed in the glass mould industry by the glass container manufacturers led in the last decade to a research and development activity that seeks to solve the various problems that have prevented a greater penetration of the glass moulds producers in the national and international market. The state of relatively low development in these companies, when this updating process began, quickly led to the conclusion that the solutions found as the main concerns of the companies created other problems, difficult to anticipate in the first analyses, that have prevented their implementation at industrial level. If surface engineering allowed to overcome and solve the rapid degradation of the surfaces of the moulding tools involved in the moulding process, which due to the direct contact with melted glass are subject to severe conditions of abrasion, corrosion, oxidation and thermal fatigue at high temperatures, it has not been able to solve (and in certain way, aggravated) the problems of temperature heterogeneity in the moulding surfaces and, consequently, the manufacture of defective glass containers. In addition, the costs involved with the use of the new mould solutions, due to both the new selected base materials and the applied coatings, created a new challenge that involves the necessary reduction of the final price of the moulds.

This study is focused on the development of surface engineering solutions based on sputtering technology – PVD, by depositing of thin films with good mechanical and tribological properties at high temperature on mould surfaces for glass industry, which could support the harsh conditions of use and increase durability relative to the state-of-the-art solutions. Within the frame of this work, transition metals were added to Titanium Nitride-based hard coatings during the deposition by DC magnetron and optimized to be with two different structure configurations: monolayer and multilayer. The coatings with multilayer architecture are an alternate option for high-temperature use, due to its low coefficient of friction and self-lubrication properties. Thus, tribological tests were performed in a tribometer at room temperature and 600°C, in order to compare

their wear rate, friction coefficient and global performance under severe conditions, allowing to predict their capability to increase tool lifetime when exposed to abrasive and corrosive environments. It's expected that the results could be a good indicator of the coatings behavior during the production process of bottles and its efficiency, thus achieving two main objectives: reduce the cost of production and increase the energy efficiency due to carbon footprint reduction.



**Figure 1: SEM image of multilayer coating produced by PVD sputtering.**

**Keywords:** Hard-coatings, glass packaging, moulds, wear resistance, energy efficiency.

## References:

1. Surface & Coatings Technology 228 (2013) 1–13



# Tool condition monitoring of self-lubricating nitride coatings to improve the lifetime of cutting tools in the manufacturing industry

J. Perdigoto <sup>1\*</sup>, B. Martins <sup>1</sup>, J. Dias <sup>1</sup>, D. Silva <sup>2</sup>, T. Todo Bom <sup>2</sup>, R. Curado <sup>2</sup>, N. Alves <sup>3</sup>, P. Rodrigues <sup>4</sup>, D. Fonseca <sup>4</sup>

<sup>1</sup> IPN- LED & MAT- Instituto Pedro Nunes, Laboratory for Wear, Testing and Materials, 3030-199 Coimbra, Portugal

<sup>2</sup> 3DTECH, Produção e Optimização de Reengenharia Lda. , 2430-528 Marinha Grande, Portugal

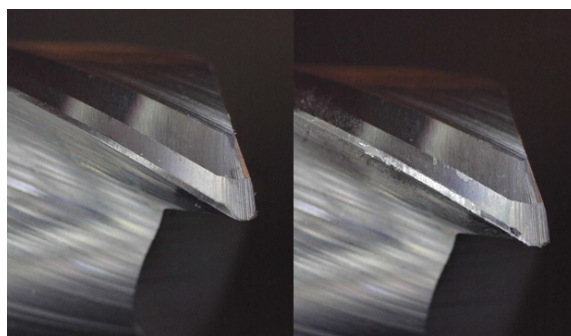
<sup>3</sup> Tj Aços, Tj Aços, Lda., 2430-528 Marinha Grande, Portugal

<sup>4</sup> NexNew, Tecnologias de Informação e Comunicação Lda. , 2415-341 Leiria, Portugal

## Abstract:

Increasing the lifetime of cutting tools involves optimizing the respective management system used in the manufacturing industry, based on chip-start cutting technology. Given the cost of producing such tools, the solution lies in the sustainability of the processes, as well as tool condition monitoring (TCM) of the wear level on the tools, reducing their environmental impact through early disposal. The rapid wear of tools during dry machining presents an opportunity for the adoption of adaptative coatings, through vacuum magnetron sputtering technologies. These coatings have wear and oxidation resistance properties and low friction at high temperatures, without lubricants during the cutting process. Ti-SiN coatings were developed with the addition of different concentrations of lubricant solid-phase agents, such as Ag or V, thus being self-lubricant coatings. For that reason, the TiSiN matrix acts as an anti-diffusion barrier of the lubricant soft phase element, preventing the rapid wear of the coating. These coatings were evaluated for their performance in dry machining tests on CK45 steel, in a laboratory environment using an automated computer control- CNC machine. Alongside these tests, the wear level of such coatings was monitored continuously and in real-time, based on optical characteristics and resistivity changes in the developed thin films. The addition of solid lubricant elements contributes to the wear reduction on cutting tools, proven by the tests carried out, thus increasing the lifetime of tools.

**Keywords:** Life cycle, Cutting tools, Stock management, Magnetron sputtering, Thin films sensorization, Tribology, Self-lubricant at high temperatures, Hard nitride coatings.



**Figure 1:** Cutting tool edge coated with Ti-SiN/TiSiVN during dry machining of CK45. Tool condition monitoring of the cutting edge to evaluate the performance of the tool.

## References:

1. Cavaleiro, D., Figueiredo, D., Moura, W., C., Cavaleiro, A., Carvalho, S., Fernandes, F., (2021) Machining performance of Ti-SiN(Ag) coated tools during dry turning of TiAl6V4 aerospace alloy, *Cer. Intern.*, 47, 11799-11806.
2. Fernandes, F., Loureiro, A., Polcar, T., Cavaleiro, A. (2014), The effect of increasing V content on the structure, mechanical properties and oxidation resistance of Ti-Si-V-N films deposited by DC reactive magnetron sputtering, *App. Surface. Science*, 8, 114-123.

# Challenges on Protein Bioencapsulation in Transparent Nanoporous CARBOTHERMAL REDUCTION OF MILL SCALES FORMED ON STEEL BILLETS DURING CONTINUOUS CASTING

S.M. Espinoza Suarez<sup>1</sup>, L.E. Borja-Castro<sup>1</sup>, M. I. Valerio-Cuadros<sup>1,2</sup>, A. Bustamante Domínguez<sup>1</sup>, H.A. Cabrera-Tinoco<sup>1,3</sup>, E. Huaman<sup>1</sup>, R.A. Valencia-Bedregal<sup>1</sup>, Xiaoli Zhao<sup>4</sup>, L. De Los Santos Valadares<sup>1,4,5</sup>.

<sup>1</sup>Laboratorio de Cerámicos y Nanomateriales, Facultad de Ciencias Físicas, Universidad Nacional Mayor de San Marcos, Ap. Postal 14-0149, Lima, Perú.

<sup>2</sup> Departamento de Física, Universidade Estadual de Maringá, Av. Colombo, 5790 - Jardim Universitário, Maringá, PR, 87020-900, Brazil.

<sup>3</sup>Facultad de Ingeniería, Universidad Continental, Lima 15311, Perú.

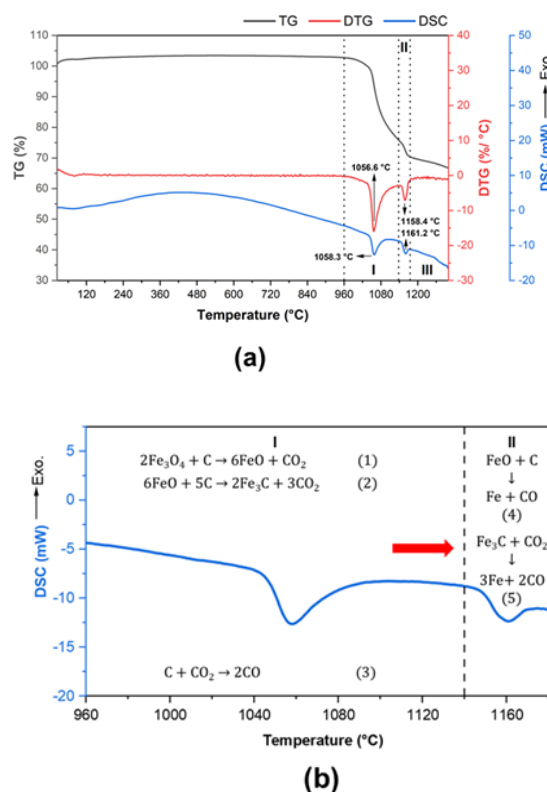
<sup>4</sup>School of Materials Science and Engineering, Northeastern University, No 11, Lane 3, Wenhua Road, Heping District, Shenyang 110819, Liaoning, People's Republic of China

<sup>5</sup>Cavendish Laboratory, Department of Physics, University of Cambridge, J.J Thomson Ave., Cambridge CB3 0HE, United Kingdom

## Abstract:

A billet is a bar made from crude steel which surface contains scales which are rich in iron oxides. This study presents the carbothermal reduction of the scales formed in steel billets. The process included the reaction of the iron oxides contents with carbon (in ratio 5:1) and annealing in a tubular furnace under argon atmosphere. The occurred reactions are discussed using thermodynamic calculations and thermal analysis which indicate a three-stage reduction process  $\text{Fe}_3\text{O}_4 \rightarrow \text{FeO} \rightarrow \text{Fe}_3\text{C} \rightarrow \alpha\text{-Fe}$  with intermediate reactions at the interval temperature 960 and 1300 °C. The X-ray diffraction confirms the reduction to  $\alpha\text{-Fe}$  with minor presence of unreacted C, magnetite and wustite. Mössbauer spectroscopy analysis was performed at room temperature where a typical sextet corresponding to the dominant  $\alpha\text{-Fe}$  is shown as well as wustite, magnetite and cementite to a lesser extent. The magnetization measurements confirm the ferromagnetic state corresponding to the  $\alpha\text{-Fe}$ .

**Keywords:** Mill scales, Steel billets, continuous casting process, steel industry, Mossbauer spectroscopy, Carbothermal reduction, X-ray diffraction.



**Figure 1:** a) Thermogram (TG – DTG/DSC) and b) zoomed DSC curve of the carbothermal reduction process of mill scales formed on steel billets. The most possible reaction equations are written per stage.

## Acknowledgment:

This work was supported by the Incorporación de Investigadores Program from the CONCYTEC – FONDECYT- UNMSM (Contract No. 12 -2019 – FONDECYT – BM – INC. INV.).

**References:**

1. Corder, G.D., Golev, A., Giurco, D.: "Wealth from metal waste": Translating global knowledge on industrial ecology to metals recycling in Australia. *Miner. Eng.* 76, 2–9 (2015). <https://doi.org/10.1016/j.mineng.2014.11.004>
2. Ghosh, A., Chatterjee, A.: *Ironmaking and Steelmaking: Theory and Practice*. (2008)
3. Bhatta, G., De Los Santos Valladares, L., Liu, X., Ma, Z., Bustamante Domínguez, A.G., Moreno, N.O., Espinoza Suarez, S.M., Barnes, C.H.W., Zhang, D.: Microstructure and mechanical properties of solid state recycled 4Cr5MoSiV (H11) steel prepared by powder metallurgy. *Results Mater.* 10, 100184 (2021). <https://doi.org/10.1016/J.RINMA.2021.100184>
4. Ubando, A.T., Chen, W.H., Ong, H.C.: Iron oxide reduction by graphite and torrefied biomass analyzed by TG-FTIR for mitigating CO<sub>2</sub> emissions. *Energy*. 180, 968–977 (2019). <https://doi.org/10.1016/j.energy.2019.05.149>
5. Shomate, C.H.: A method for evaluating and correlating thermodynamic data, <https://pubs.acs.org/doi/abs/10.1021/j150514a018>, (1954)
6. Chase, J.M.W.: *NIST-JANAF Thermochemical Tables-Fourth edition*. *J. Phys. Chem. Ref. Data*. monograph, 1–1951 (1998)
7. Treptow, R.S., Jean, L.: The Iron Blast Furnace: A Study in Chemical Thermodynamics. *J. Chem. Educ.* 75, 43 (1998). <https://doi.org/10.1021/ed075p43>
8. Mondal, K., Lorethova, H., Hippo, E., Wiltowski, T., Lalvani, S.B.: Reduction of iron oxide in carbon monoxide atmosphere—reaction controlled kinetics. *Fuel Process. Technol.* 86, 33–47 (2004). <https://doi.org/https://doi.org/10.1016/j.fuproc.2003.12.009>
9. Borja-Castro, L.E., Bustamante Domingues, A.G., Valerio-Cuadros, M.I.: Characterization of steel billet scales generated during the continuous casting process in SIDERPERU. (2021)
10. Sheshukov, O., Mikheenkoy, M., Vedmid', L., Nekrasov, I., Egiazaryan, D.: Mechanism of Ion-Diffusion Solid-Phase Reduction of Iron Oxides of Technogenic Origin in the Presence of the Liquid Phase and without it. *Metals (Basel)*. 10, 1564 (2020). <https://doi.org/10.3390/met10121564>
11. Kawanari, M., Matsumoto, A., Ashida, R., Miura, K.: Enhancement of reduction rate of iron ore by utilizing iron ore/carbon composite consisting of fine iron ore particles and highly thermoplastic carbon material. *ISIJ Int.* 51, 1227–1233 (2011). <https://doi.org/10.2355/isijinternational.51.1227>
12. Chen, Z., Dang, J., Hu, X., Yan, H.: Reduction kinetics of hematite powder in hydrogen atmosphere at moderate temperatures. *Metals (Basel)*. 8, (2018). <https://doi.org/10.3390/met8100751>
13. Treptow, R.S., Jean, L.: The iron blast furnace: A study in chemical thermodynamics. *J. Chem. Educ.* 75, 43–47 (1998). <https://doi.org/10.1021/ed075p43>
14. Jozwiak, W.K., Kaczmarek, E., Maniecki, T.P., Ignaczak, W., Maniukiewicz, W.: Reduction behavior of iron oxides in hydrogen and carbon monoxide atmospheres. *Appl. Catal. A Gen.* 326, 17–27 (2007). <https://doi.org/10.1016/j.apcata.2007.03.021>
15. Kramm, U.I., Ni, L., Wagner, S.: 57Fe Mössbauer Spectroscopy Characterization of Electrocatalysts. *Adv. Mater.* 31, 1–11 (2019). <https://doi.org/10.1002/adma.201805623>
16. Stevens, J.G., Khasanov, A.M., Miller, J.W., Pollak, H.: *Mossbauer Mineral Handbook*. (2002)
17. Gheisari, M., Mozafari, M., Niyafar, M., Amighian, J., Soleimani, R.: Observation of small exchange bias in defect wüstite (Fe<sub>0.93</sub>O) nanoparticles. *J. Supercond. Nov. Magn.* 26, 237–242 (2013). <https://doi.org/10.1007/s10948-012-1821-9>
18. Cho, S., Lee, J.: Metal recovery from stainless steel mill scale by microwave heating. *Met. Mater. Int.* 14, 193–196 (2008). <https://doi.org/10.3365/met.mat.2008.04.193>
19. Vandenberghe, R.E., Barrero, C.A., Da Costa, G.M., Van San, E., De Grave, E.: Mössbauer characterization of iron oxides and (oxy)hydroxides: The present state of the art. *Hyperfine Interact.* 126, 247–259 (2000). <https://doi.org/10.1023/A:1012603603203>

# Study of Mill Scale Surface Morphology and Iron Oxide Layers Obtained During Quenching of Steel Billets in The Continuous Casting

R. A. Valencia-Bedregal<sup>1\*</sup>, L. E. Borja-Castro<sup>1</sup>, M. I. Valerio-Cuadros<sup>1,2</sup>, S. M. Espinoza Suarez<sup>1</sup>, A. G. Villasante Miranda<sup>1,3</sup>, E. Y. Huaman Enriquez<sup>1,3</sup>, A. Bustamante Dominguez<sup>1</sup>, L. De Los Santos Valladares<sup>1,4</sup>

<sup>1</sup> Laboratorio de Cerámicos y Nanomateriales, Facultad de Ciencias Físicas, Universidad Nacional Mayor de San Marcos, Ap. Postal 14-0149, Lima, Peru

<sup>2</sup> Departamento de Física, Universidade Estadual de Maringá, Av. Colombo, 5790 – Jardim Universitário, Maringá, PR 87020-900, Brazil

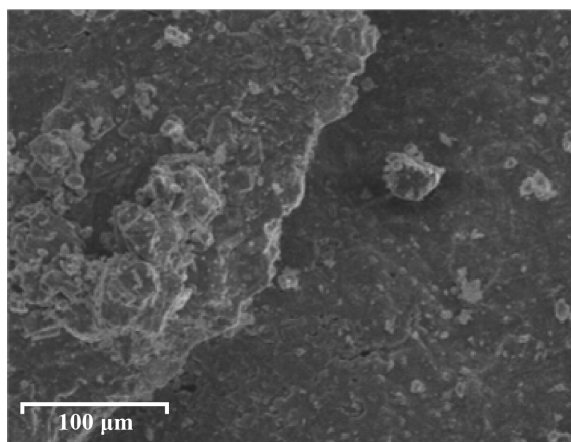
<sup>3</sup> Escuela de Ingeniería Metalúrgica, Universidad Nacional Mayor de San Marcos, Ap. Postal 14-0149, Lima, Peru

<sup>4</sup> Cavendish Laboratory, Department of Physics, University of Cambridge, J.J Thomson Av., Cambridge CB03 0H3, UK

## Abstract:

In the last step of the steelmaking process, known as continuous casting, the mill scale is formed on the surface of the steel billet during the quenching. The mill scales present an exterior layer of hematite, an intermediate layer of magnetite and an inner layer of non-stoichiometric wüstite which is the predominant phase [1,2]. The study of mill scales surface morphology and layers are important to understand the iron oxides formed on the surface of the steel. This work presents the advances in the surface analysis of mill scales, in the image taken by Scanning Electron Microscope (SEM) reveals particles about 100  $\mu\text{m}$  diameter and the morphology of the mill scales present a rough surface (Figure 1). The elemental composition by Electron Dispersive X-ray and X-ray Fluorescence shows Fe (72.8 wt%, 97.9 %) as the major constituent and other elements such as Mn, Co, Cu, Si and Ca in minor concentration ( $\sim <1\%$ ) [3]. The crystallographic phases detected by X-ray Diffraction are, as mentioned above, wüstite as the principal phase followed by magnetite and hematite. Other technics are applied to complement the study are Mossbauer Spectroscopy and Magnetometry measurements.

**Keywords:** Mill scales, Steel billet, Continuous Casting, Iron Oxides, SEM, EDS, XRF, XRD.



**Figure 1:** SEM micrograph showing particles about 100  $\mu\text{m}$  diameters and the morphology of the mill scales present a rough surface, the sample was obtained during quenching of steel billets in the continuous casting.

## References:

1. Bagatini, M. C., Zymła, V., Osório, E., & Vilela, A. C. F. (2011). Characterization and reduction behavior of mill scale. *Isij International*, 51(7), 1072-1079.
2. Yusupkhodjaev, A. A., Samadov, A. U., Khojiev Sh, T., Nuraliev, O. U., & Ubaydul-laeV, A. U. (2020). Study of iron extraction from mill scale.
3. Borja-Castro, L. E., Bustamante Domingues, A. G., & Valerio-Cuadros, M. I. (2021). Characterization of steel billet scales generated during the continuous casting process in SIDERPERU.

# Surface and structural characterization of recycled HRB335 steel from black slags obtained from Electric Arc Furnace

E.Y. Huaman Enriquez<sup>1,2</sup>, S.M Espinoza Suarez<sup>1</sup>, A.G. Villasante Miranda<sup>1,2</sup>, L.E. Borja-Castro<sup>1</sup>, R.A. Valencia-Bedregal<sup>1</sup>, M.I. Valerio-Cuadros<sup>1,3</sup>, A. Bustamante Domínguez<sup>1</sup>, Xiaoli Zhao<sup>4</sup>, Deliang Zhang<sup>3</sup>, L. De Los Santos Valladares<sup>1,4,5</sup>

<sup>1</sup> Laboratorio de Cerámicos y Nanomateriales, Facultad de Ciencias Físicas, Universidad Nacional Mayor de San Marcos, Perú

<sup>2</sup> Escuela de Ingeniería Metalúrgica, Universidad Nacional Mayor de San Marcos, Lima, Perú

<sup>3</sup> Departamento de Física, Universidade Estadual de Maringá, Av. Colombo, 5790 – Jardim Universitário, Maringá, PR, 87020-900, Brazil

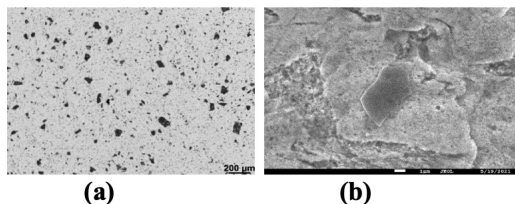
<sup>4</sup> School of Materials Sciences and Engineering, Northeastern University, No 11, Lane 3, Wenhua Road, Heping District, Shenyang 110819, People's Republic of China.

<sup>5</sup> Cavendish Laboratory, Department of Physics, University of Cambridge, Cambridge CB03 0HE, UK

## Abstract:

Due to the structural suitability of HRB335 steel commonly used in the construction industry [1], it is necessary to perform a superficial and structural analysis to determine the presence of irregularities which can develop stress concentration and preferential spots for corrosion, generating a decrease in its mechanical properties [2–4]. It is necessary to mention that this steel has a particular processing since it has been produced from the black slag of an electric arc furnace through carbon reduction processes [5] and powder metallurgical shaping by extrusion and sintering. Therefore, physical characteristics such as the degree of porosity and second phase components must be considered. These HRB335 steel rods were measured using X-ray diffraction and Mössbauer spectroscopy [6, 7]. The results show that  $\alpha$ -Fe is the main crystallographic phase revealed by X-ray diffraction and  $^{57}\text{Fe}$  Mössbauer spectroscopy taken at RT and 50 K. Additional studies include Scanning Electron Microscopy and Optical Micrography.

**Keywords:** HRB335 steel, steel rods, SEM, Optical micrography, X-ray diffraction, Mössbauer spectroscopy



**Figure 1**  
Microstructure of the cross section of HRB335 steel rod, (a) Ferrite matrix with

amount of second phase in Optical micrograph image. (B) Second phase containing carbon, carbide, Mn, and Si in SEM analysis.

## References:

1. Lin, F., Dong, Y., Kuang, X., Lu, L.: Strain rate behavior in tension of reinforcing steels HPB235, HRB335, HRB400, and HRB500. *Materials* (Basel). 9, (2016). <https://doi.org/10.3390/ma9121013>
2. Shi, W. ning, Yang, S. feng, Li, J. she: Effect of nonmetallic inclusions on localized corrosion of spring steel. *Int. J. Miner. Metall. Mater.* 28, 390–397 (2021). <https://doi.org/10.1007/s12613-020-2018-z>
3. Meurling, F., Melander, A., Tidesten, M., Westin, L.: Influence of carbide and inclusion contents on the fatigue properties of high speed steels and tool steels. *Int. J. Fatigue.* 23, 215–224 (2001). [https://doi.org/10.1016/S0142-1123\(00\)00087-6](https://doi.org/10.1016/S0142-1123(00)00087-6)
4. Qi, J., Halloran, J.W.: Negative thermal expansion artificial material from iron-nickel alloys by oxide co-extrusion with reductive sintering. *J. Mater. Sci.* 39, 4113–4118 (2004). <https://doi.org/10.1023/B:JMSC.0000033391.65327.9d>
5. Suarez, S.M.E., Borja-Castro, L.E., Valerio-Cuadros, M.I., Domínguez, A.B., Cabrera-Tinoco, H.A., Huaman, E., Valencia-Bedregal, R.A., Zhao, X., Zhang, Y., Zhang, D., Barnes, C.H.W., Valladares, L.D.L.S.: Carbothermal reduction of mill scales formed on steel billets during continuous casting.

- Hyperfine Interact. 242, (2021).  
<https://doi.org/10.1007/s10751-021-01769-9>
6. Greenwood, N.N., Gibb, T.C.: Mössbauer Spectroscopy. Springer Netherlands (1971)
  7. Swanson: Iron Card- Natl Bur Stand U.S. Cir. (1955)



# Surface morphology of an Extruded Q235 Steel Rod Fabricated from Rolling Billets Scales

A. G. Villasante Miranda<sup>1,2\*</sup>, E. Y. Huaman Enriquez<sup>1,2</sup>, L.E. Borja Castro<sup>1</sup>, R. Valencia Bedregal<sup>1</sup>, S.M. Espinoza Suarez<sup>1</sup>, M.I. Valerio Cuadros<sup>1,3</sup>, Xiaoli Zhao<sup>4</sup>, A. Bustamante Dominguez<sup>1</sup>, L. De Los Santos Valladares<sup>1,4,5</sup>

<sup>1</sup>Laboratorio de Cerámicos y Nanomateriales, Universidad Nacional Mayor de San Marcos, Lima, Perú

<sup>2</sup>Escuela de Ingeniería Metalúrgica, Universidad Nacional Mayor de San Marcos, Lima, Perú

<sup>3</sup>Departamento de Física, Universidade Estadual de Maringá, Maringá, Brazil

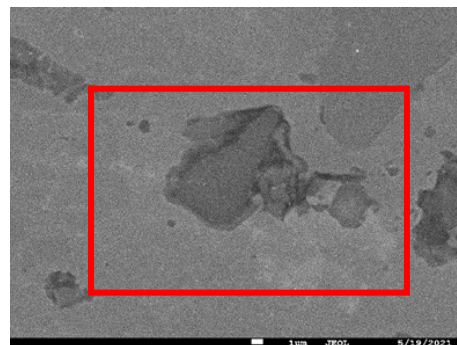
<sup>4</sup>School of Materials Science and Engineering, Northeastern University, Shenyang 110819, People's Republic of China

<sup>5</sup>Cavendish Laboratory, Department of Physics, University of Cambridge, Cambridge CB03 0HE, UK

## Abstract:

Steel is one of the most common material used in engineering since due to its applications and interesting properties such as hardness, wear resistance, corrosion resistance, ductility and others. In addition to the bulk properties, its surface morphology also plays a role during the steel applications (e.g. corrosion, oxidation resistance and so on). During the last years the demand of to produce steel has been increased. For that reason, recycling wastes from steelmaking process such as dust, oxides, scales and slags result has been turned to an important alternative to elaborate new steel [1,2]. For example, during the rolling process of steel, oxide scales are produced in the surface of the billets due to oxidation at high temperatures. We have collected this rolling billets scales, reduced its Fe content and elaborated new Q235 steel rods. Q235 steel presents qualified properties that make it a good option for its applications, some of their properties are good corrosion resistance, good tenacity, good resistance and good weldability [1,3,4]. In this work, we characterized the surface morphology of the recycled Q235 steel rod in order to find possible pores, oxides and impurities that can affect the properties and performance rod. Figure 1 shows a SEM (Scanning Electron Microscopy) micrograph presenting an inclusion known as silicide. This type of inclusion improves the resistance of the steel without affecting significantly the ductility [5]. Addition analysis include metallography, fractography, X-ray diffraction and Mössbauer spectroscopy.

**Keywords:** Q235 steel, metallography, SEM, fractography, X-ray diffraction, Mössbauer spectroscopy



**Figure 1:** Photo taken using SEM technique. The phase in the red rectangle corresponds to the presence of silicide in the Q235 steel rod elaborated from rolling billet scales

## References:

1. Villasante Miranda, A.G., Borja Castro, L.E., Valencia-Bedregal, R.A., Espinoza Suarez, S. M., Valerio-Cuadros, M. I., Bustamante Domínguez, A., Zhao, X., Zhang, Y., Zheng, C., Barnes, C. H. W., Zhang, D., De Los Santos Valladares L., (2021) Characterization of recycled Q235 steel chips from rolling billets scales. *Hyperfine Interactions*, 242(1)
2. Makkonen, H.T., Heino, J., Laitila, L., Hiltunen, A., Pöylyö, E., Härkki, J. (2002) Optimisation of steel plant in Finland: dusts, scales and slugs. *Resour. Conserv. Recycl.* 35, 77-84
3. Shi, G., Wang, S., Chen, C., Rong, C. (2021) Post-fire mechanical properties of base metal and welds of Q235 steel. *J. Constr. Steel Res.* 183, 106767.
4. Wu, Y.H., Liu, T.M., Luo, S. X., Sun, C. (2010) Corrosion characteristics of Q235 steel simulated Yingtan soil solutions. *Materwiss. Werksttech* 41, 142-146
5. D. Bowden, Y. Krysiak, L. Palatinus, D. Tsivoulas, S. Plana-Ruiz, E. Sarakinou, U. Kolb, D. Stewart, M. Preuss (2018) A high-strength silicide phase in a stainless steel alloy designed for wear-resistant applications. *Nat. Commun* 9, 1-10

## Morphological and Mineral characterization of Mill Scales obtained by rolling process

L.E. Borja Castro<sup>1</sup>, J. Kargin<sup>2</sup>, R.A. Valencia Bedregal<sup>1</sup>, S.M Espinoza Suarez<sup>1</sup>, A.G. Villasante Miranda<sup>1</sup>, M.I. Valerio-Cuadros<sup>1,3</sup>, A. Bustamante Domínguez<sup>1</sup>, L. De Los Santos Valladares<sup>1,4</sup>

<sup>1</sup> Laboratorio de Cerámicos y Nanomateriales, Facultad de Ciencias Físicas, Universidad Nacional Mayor de San Marcos, Perú

<sup>2</sup> Department of Technologies Commercialization, L.N. Gumilyov Eurasian National University, 010000 Astana, Kazakhstan

<sup>3</sup> Departamento de Física, Universidade Estadual de Maringá, Av. Colombo, 5790 – Jardim Universitário, Maringá, PR, 87020-900, Brazil

<sup>4</sup> Cavendish Laboratory, Department of Physics, University of Cambridge, Cambridge CB03 0HE, UK

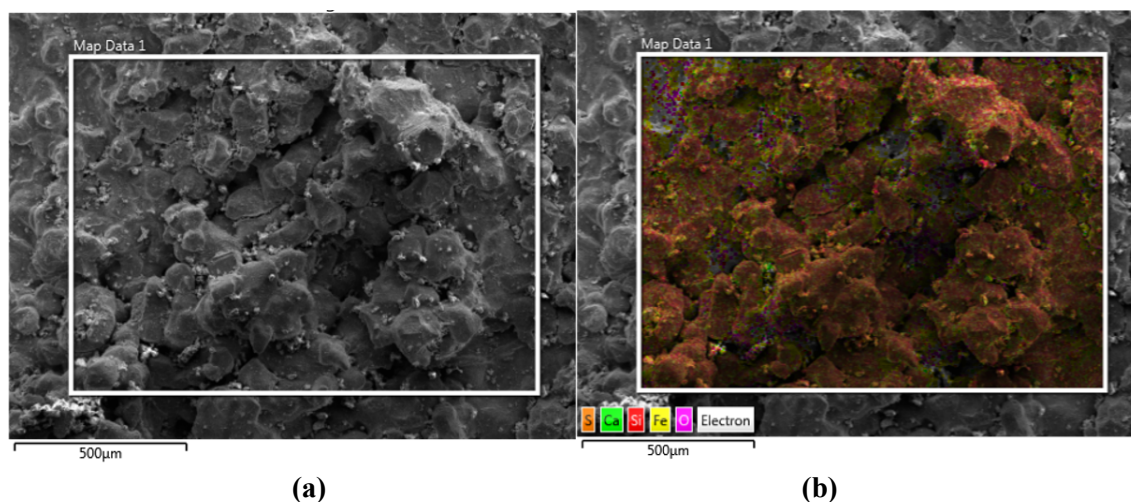
### Abstract:

Mill scale is a by-product of steel generated by the final processes of steel production as a product of surface oxidation of steel due to a thermal gradient, a corrosive environment or the simple action of time [1, 2]. In this work we study the morphology and mineralogy of mill scale generated during the hot rolling process of steel in order to understand their formation on steel surfaces. Adapted scanning electron microscopy with

energy dispersive X-rays (SEM-EDX) detects as main chemical elements Fe and O; and Si, S, Ca, Mg, C and Al as the minimum elemental composition. The XDR measurements reveals  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> (hematite) and Fe<sub>3</sub>O<sub>4</sub> (magnetite) as the predominant mineralogical phases. Additional measurements such as Mössbauer Spectroscopy reveals the presence of wustite.

**Keywords:** Mill Scales, Oxidation, Morphology, Steel.

**Figure 1**



SEM micrographs of mill scales obtained by industrial rolling mill steel: (a) morphology and (b) EDX mapping.

### References:

1. Cunha, A.F. da, Mol, M.P.G., Martins, M.E., Assis, P.S.: Caracterização, beneficiamento e reciclagem de carepas geradas em processos siderúrgicos. Rem Rev. Esc. Minas. 59, 111–116 (2006). <https://doi.org/10.1590/s0370-44672006000100014>
2. Prim, S.R., Folgueras, M. V., de Lima, M.A., Hotza, D.: Synthesis and characterization of hematite pigment obtained from a steel waste industry. J. Hazard. Mater. 192, 1307–1313 (2011). <https://doi.org/10.1016/j.jhazmat.2011.06.034>

## Hydrophobic concrete based on hydrophobic sand

Seralin A., Kurbanova A., Khassenova T., Ualibek O., Sugurbekova G., Toktarbaiuly O.  
National Laboratory Astana, Nur-Sultan, Kazakhstan

### Abstract

One of the main enemies of any concrete in environmental conditions, affecting on its properties and durability is the water. The rainwater from acidic rains may penetrate inside of the structure and expose the concrete to more early degradation. That is why there are exist a series of methods how to protect the concrete from water action.

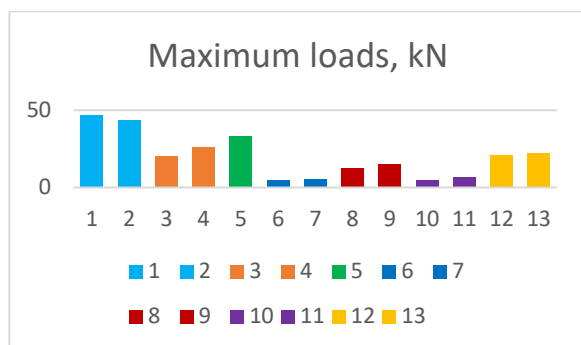
In this research, we synthesized the hydrophobic sand via patented technology and implemented the concrete forms in order to obtain hydrophobic concrete stable not to absorb the water and moisture. For comparison we also prepared several samples based on other hydrophobizators like silicon oil, motor oil and measured the strength of obtained forms.



a) treated hydrophobic sand used



b) regular sand used



Sample №	Max. loads, kN	Description
1*	46.75	Control (cement+sand+water) *
2	43.83	Control (cement+sand+water)
3*	20.14	Silicon oil *
4	25.96	Silicon oil
5*	33.04	Hydrophobic sand *
6*	4.99	Rubber+Silicon oil *
7	5.74	Rubber+Silicon oil
8*	12.39	Motor oil (non-used) *
9	14.99	Motor oil (non-used)
10*	4.72	Used motor oil, 5% *
11	7.11	Used motor oil, 5%
12*	21.06	Used motor oil, 2.5% *
13	22.2	Used motor oil, 2.5%

\* - sample was treated during 2 hours in water and hold 20 h at -30 °C



# A microfluidic non-thermal plasma reactor equipped with inline emission spectroscopy.

P. Roszkowska<sup>1</sup>, A. Slater<sup>1</sup>, J. Walsh<sup>2</sup>, A. Dickenson<sup>2</sup>, T. Easun<sup>3</sup>

<sup>1</sup> Department of Chemistry, University of Liverpool, Liverpool, UK

<sup>2</sup> Centre for Plasma Microbiology, Department of Electrical Engineering and Electronics, University of Liverpool, Liverpool, UK

<sup>3</sup> School of Chemistry, Cardiff University, Cardiff, CF10 3XQ, UK

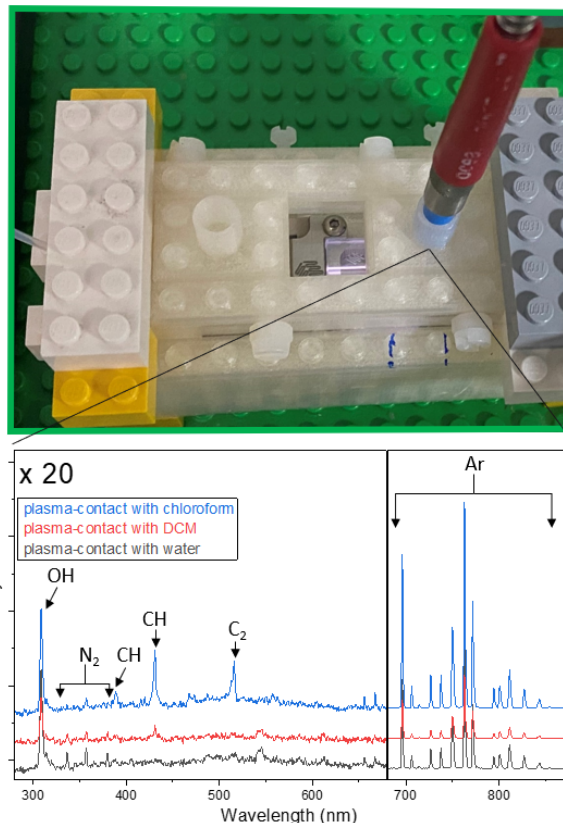
## Abstract:

Every chemical reaction relies on electrons: supplying a controlled source of energetic electrons is a fundamental route to controlled chemical transformations. Non-Thermal Plasma (NTP) is a source that provide with high energy electrons and other reactive species whilst remaining at moderate temperature.[1] If NTP could be produced and mixed with a liquid in a controlled way it would be ideal for assisting/conducting chemical reactions. However, many current methods of studying interactions between plasma and liquid do not give sufficient time or spatial resolution to track and identify the complex and short-lived species involved.[2, 3] Additionally, many existing NTP reactor designs have drawbacks such as solvent evaporation and lack a profound understanding of the underpinning mechanisms responsible for the observed chemical reactions.[4]. Also, the majority of studies about NTP-liquid interaction were done with water, which severely restricts the applicability of the findings for chemistry purposes.[5] Here, we detail the construction of a microfluidic reactor capable of running chemical reactions using NTP. In our experiments we observed NTP in contact with dichloromethane (DCM), chloroform and water. We recorded the spectra emitted by plasma in contact with those solvents using microfluidic chip with in-situ Optical Emission Spectroscopy (OES) device. Furthermore, we designed a setup that allows batch comparison of any study performed in our microfluidic setup. The batch device is very easily setup and allows for reactions under inert atmosphere.

This study illustrates the vast potential that NTP can offer in chemical reaction. Our next step is to evaluate a range of selected chemical reactions to determine how they can benefit from NTP technology.

**Keywords:** Non thermal plasma, chemistry, microfluidics, green chemistry, non thermal plasma in contact with liquid, non thermal plasma liquid activation, OES, NTP technology.

Catalytic reactions with plasmas/Plasmas in (contact with) liquids / plasma-liquid interaction/Non thermal plasma chemis



**Figure 1:** Figure illustrating the NTP microfluidic reactor with in-situ OES device (top) and emission spectra of plasma on contact with chloroform, DCM and water.

## References:

1. Tendero C, Tixier C, Tristant P, et al (2006) Atmospheric pressure plasmas: A review. *Spectrochim. Acta - Part B* 61:2–30
2. Palma V, Cortese M, Renda S, et al (2020) A review about the recent advances in selected nonthermal plasma assisted solid–gas phase chemical processes. *Nanomaterials*
3. Wright A, Taglioli M, Montazersadgh F, et al (2019) Microbubble-enhanced DBD plasma reactor: Design, characterisation and modelling. *Chem Eng Res Des* 144:159–173. 0
4. Gorbanev Y, Leifert D, Studer A, et al (2017) Initiating radical reactions with non-thermal

- plasmas. *Chem Commun* 53:3685–3688.
5. Adamovich I, Baalrud SD, Bogaerts A, et al (2017) The 2017 Plasma Roadmap: Low temperature plasma science and technology. *J. Phys. D. Appl. Phys.* 50

# Remotion of the degraded coating of the polymer injection moulds for their reuse.

R. Santos<sup>1</sup>, T. Ferreira<sup>2</sup>, L. Pereira<sup>3</sup>, F. Costa<sup>1</sup>, N. M. Ferreira<sup>1</sup>

<sup>1</sup> i3N & Physics Department, University of Aveiro, Aveiro, Portugal

<sup>2</sup> IPN, Coimbra, Portugal

<sup>3</sup> MicroPlasticos, Gala, Figueira da Foz, Portugal

## Abstract:

In the plastic/polymers injection industry, the moulds must be in perfect condition to secure efficient production. However, the self-lubricating coating thickness is expected to reduce with the continued manufacture of pieces, degrading the functional surface of the moulds. For the reuse of the mould substrate, a new functional coating will be needed to be deposited, increasing the mould lifetime. Still, this new coating deposition can only occur after the remotion of the degraded one.

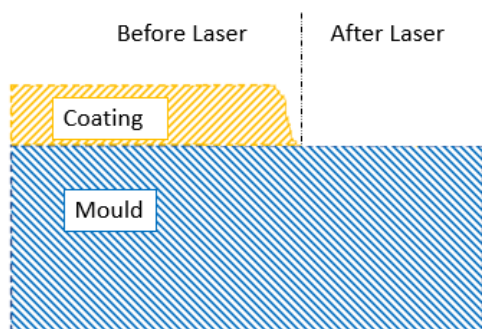
Several examples of using laser technology to remove coating could be found. Yet, the majority of these coating is paint or oxide.

A study of the implementation of the laser technology to remove the functional coating of a mould was addressed, (Fig.1) the coating is the type of TiN, that absorbs radiation of Fiber laser. The optimization of the laser parameters such as power, frequency, scan speed, etc, is required to remove efficiently the coating and prepare the substrate, like their surface roughness, to receive the new coating by PVD, without modifying the dimensions of the mould or else the pieces produced by those could not pass by the quality control checks. The results show the efficiency of the first trials to prove the concept for coating removal without damaging the mould.

**Keywords:** Fiber Laser; functional coating removal; injection moulds, roughness, PDV deposition, coating removal

## References:

1. D. E. Roberts, Pulsed laser coating removal by detachment and ejection, *Applied Physics A* 79 (2004) 1067-1070.
2. M. Shamsujjoha et al., Effects of laser ablation coating removal (LACR) on a steel substrate: Part 1: Surface profile, microstructure, hardness, and adhesion; *Surface and Coating Technology*; 281 (2015) 193-205.
3. <https://cleanmould.web.ua.pt/wp>



**Figure 1:** Figure illustrating of the methodology for the coating removal, by laser.