SICT 2024, PLASMA TECH 2024 AND TRIBOLOGY 2024

JOINT INTERNATIONAL CONFERENCES

17 - 19 APRIL 2024, VIENNA - AUSTRIA

Book of Abstracts

Organizer

SETCOR
Conferences & Exhibitions

www.setcor.org

SICT 2024 / Plasma Tech 2024 / Tribology 2024 Joint Conferences Program

17 – 19 April 2024 | Vienna, Austria

17 April 2024		
09:00 - 12:00	**Participants registration**	
09:30 - 10:30	**Welcoming Coffee**	
SICT 2024 / Plasma Tech 2024 / Tribology 2024 Joint Plenary Session		
Conference Room: Salon Schönbrunn		
Session's Chairs: **Prof. Holger Kersten, University Kiel, Germany** **Prof. Miran Mozetic, Jozef Stefan Institute, Slovenia**		
10:30 - 11:00	Plasma Chemical Surface Engineering **D. Hegemann** and P. Navascués	**Prof. Dirk Hegemann**, Swiss Federal Laboratories for Materials Science and Technology, **Switzerland**
11:00 - 11:30	Diagnostic of plasma processes for electric space propulsion **H. Kersten**, T. Trottenberg, V. Schneider, A. Spethmann, R. Wimmer-Schweingruber	**Prof. Holger Kersten**, University Kiel, **Germany**
11:30 - 12:00	Introduction of Anodic Oxide Film Formation Technology of Light Metals **S. Moon**	**Prof. Sungmo Moon**,Korea Institute of Materials Science & University of Science & Technology, **Rep. of Korea**
12:00 - 14:00	**Lunch Break – Restaurant**	
SICT 2024 Session I. A: **Surface treatments and coatings deposition and functionalization / Characterization / Properties** **Multifunctional composite and hybrid coatings**		
Conference Room: Salon Schönbrunn 2 / 3		
Session's Chairs: **Prof. Stanko Brankovic, University of Houston, USA** **Prof. Sungmo Moon,Korea Institute of Materials Science, Rep. of Korea** **Prof. Giovanna Trevisi, CNR, Italy**		
14:00 - 14:30	Structural Modification of Electrodeposited Cu 3D Structures for the Enhancement of CO2 Cathodic Reduction B. Serapiniene, A. Selskis, L. Gudaviciute, J. Juodkazyte and **R. Ramanauskas**	**Prof. Rimantas Ramanauskas**, Center for Physical Sciences and Technology, **Lithuania**
14:30 - 14:45	Novel Industrial Sputtering Technology with Enhanced Flux Fraction of Deposited Material **J. Klusoň**, M. Učík, M. Jílek, J. Hnilica, P. Klein, P. Vašina, H. Bolvardi and A. Lümkemann	**Dr.Jan Kluson**, PLATIT a.s., **Czech Rep.**
14:45 - 15:00	Chemical vapour deposited SiOxNyCz thin Films for Advanced Barrier Solutions: Probing Chemical and Mechanical properties for better Performance **F. Inoubli**, B. Diallo, K. C. Topka, T. Sauvage, R. Laloo, B. Caussat, V. Turq and N. Pellerin	**Ms. Farah Inoubli**, University of Orleans, **France**
15:00 - 15:15	The impact of growth conditions on the crystalline quality and surface morphology of epitaxial III-N thin films: A molecular dynamics study **A. Zaiter**, V. Hounkpati, G. Clavier, P. Ruterana and J. Chen	**Mr. Ayla Zaiter**, Caen Normandie University, **France**
15:15 - 15:30	Chitosan Functionalized Hexagonal Boron Nitride Nano sheets as Reinforcements for the Improvement of Anticorrosive Epoxy Coatings on AA2024 Al Alloy in Saline Medium **A.Madhan Kumar**	**Dr. Madhan Kumar Arumugam**, King Fahd University of Petroleum & Minerals, **Saudi Arabia**
15:30 - 15:45	Enthalpy-driven self-healing in thin metallic films on flexible substrates	**Dr. Claus Trost,** Erich Schmid Institute of Materials

	C.O.W. Trost, P. Kreiml, T. Jörg, V. Terziyska, C. Mitterer, M. J. Cordill and A. Lassnig	Science of the Austrian Academy of Sciences, **Austria**
15:45 - 16:00	Comparison of nitriding behaviour for austenitic and duplex stainless steel with precipitation hardening steel D. Manova and **S. Mändl**	**Dr. Stephan Mändl**, Leibniz Institute of Surface Engineering, **Germany**
16:00 - 16:30	**Coffee Break / Posters Session**	
16:30 - 16:45	Challenges on Surface Oxidation of Steel – Alloying Elements and Scale Behavior **K. Anand**, A. Koltsov, L. Turri, J. Scheid, P. Alexandre	**Dr. Kanika Anand**, ArcelorMittal R&D Maizieres, **France**
16:45 - 17:00	Influence of current density and process time on structural, chemical and physical behaviour of anodized aluminum **L. Breu**, M. Bubrin and T. Sörgel	**Ms. Lea Breu**, Robert Bosch Manufacturing Solutions GmbH, **Germany**
17:00 - 17:15	Adhesion improvements of paint on polypropylene copolymers by flame and UV-ozone surface treatments **M. Borg**, Y. Tison, K. Lavisse and C. Derail	**Ms. Marine Borg**, Renault Group, **France**
17:15 - 17:30	Transparent PDMS Surfaces with Covalently Attached Lubricants for Enhanced Anti-Adhesion Performance **T. Eder**, A. Mautner, Y. Xu, M. Reithofer, A. Bismarck and J.M. Chin	**Ms. Tanja Eder**, University of Vienna, **Austria**
17:30 - 17:45	Investigation of Thermal and Mechanical Properties of Ti-Si-B-C Nanocomposite Coatings **A. Thewes**, P.M. Reinder, H. Paschke, T. Brückner, C. Sternemann, M. Paulus, W. Tillmann, J. Urbanczyk and N.F. Lopas Dias	**Mr. Alexander Thewes**, TU Braunschweig, **Germany**
17:45 - 18:00	Core/shell Ge/Al quantum dot lattices in amorphous SiC: structure and photo-conversion properties **M. Tkalcevic**, I. Periša, M. Bubaš, S. Bernstorff, G. Provatas and M. Mičetić	**Dr. Marija Tkalcevic**, Ruđer Bošković Institute, **Croatia**

17 April 2024

Plasma Tech Session I. B:
Plasma fundamentals / Modelling / Atomic and Molecular Processes

Conference Room: Salon Bristol

Session's Chairs:
Prof. Dirk Hegemann, Swiss Federal Laboratories for Materials Science and Technology, Switzerland
Prof. Holger Kersten, University Kiel, Germany

14:00 - 14:30	Plasma devices for satellite propulsion **J. Schein**	**Prof. Jochen Schein,** University of Federal Armed Forces Munich, **Germany**
14:30 - 14:45	Langmuir probe measurements in a dual-frequency capacitively coupled rf discharge **V. Schneider**, J. Schleitzer, I. Korolov G. Hübner, P.Hartmann, J. Schulze and H. Kersten	**Dr. Viktor Schneider**, University Kiel, **Germany**
14:45 - 15:00	Optically trapped microparticles in a dual-frequency capacitively coupled rf discharge **J. Schleitzer**, V. Schneider and H. Kersten	**Ms. Jessica Schleitzer**, University Kiel, **Germany**
15:00 - 15:15	A novel test-facility based on X-ray imaging and mm-wave polarimetry for plasma diagnostics **E. Naselli**, G. Finocchiaro, G.S. Mauro, B. Mishra, B. Peri, A. Pidatella, G. Torrisi and D. Mascali	**Dr. Eugenia Naselli**, National Institute for Nuclear Physics-Laboratori Nazionali del Sud (INFN-LNS), **Italy**
15:15 - 15:30	Plasma Generation and Expansion in the Vacuum Arc Remelting (VAR) process **E. Karimi-Sibaki**, A. Kharicha and C.-Doppler	**Dr. Ebrahim Karimi Sibaki**, Montanuniversitaet of Leoben, **Austria**
15:30 - 15:45	Simulation of an ion beam extraction from an ICP by using a self-consistent plasma sheath model. **K. M. Rettig**, T. Dunger, E. Loos, M. Nestler and J. Schuster	**Mr. Kevin Michael Rettig**, scia Systems GmbH, **Germany**
15:45 - 16:00	Application of high power ns-pulsed plasma for methane reforming M. Gromov, Georgios D. Stefanidis, R. Morent and **A. Nikiforov**	**Dr. Anton Nikiforov**, Ghent University, **Belgium**
16:00 - 16:30	**Coffee Break / Posters Session**	

Session's Chairs:
Prof. Jochen Schein, University of Federal Armed Forces Munich, Germany
Prof. Lado Filipovic, TU Wien, Austria

16:30 - 16:45	On the role of reaction mechanisms during the plasma-assisted dry reforming of methane **M. Oliva-Ramírez**, P. Navascués, J. Cotrino, A. R. González-Elipe and A. Gómez-Ramírez	**Dr. Manuel Oliva**, University of Seville, **Spain**
16:45 - 17:00	Silicon Carbide for neutron diagnostic in plasma **S. De Luca**, C. Altana, S. Amaducci, L. Cosentino and S. Tudisco	**Dr. Saverio De Luca**, INFN-LNS, **Italy**
17:00 - 17:15	A New Generation of Microreactor for Gas/Liquid Plasma Chemical Processes : Study of Segmented Flow under Plasma **P. Dedieu**, G. Morand, S. Ognier and M. Tatoulian	**Mr. Pierre Dedieu**, Institut de Recherche de Chimie de Paris, PSL, **France**
17:15 - 17:30	Plasma-catalytic hybrid process for CO_2 valorization into liquid fuels **N. Merino**, S. Ognier, X. Duten, M. Mikhail and M. Tatoulian	**Mrs. Noelia Merino**, Institut de Recherche de Chimie Paris – ENERGO, **France**
17:30 - 17:45	Degradation of organic pollutants in water through plasma - catalysis process D. Tsokanas, O. Dolinski, S. Meropoulis and **C. Aggelopoulos**	**Dr. Christos Aggelopoulos**, Foundation for Research and Technology Hellas, **Greece**
17:45 - 18:00	Electron emission Electron Emission Yield of Cu, and Au: Effects of Electron Irradiation and Argon Etching **M. Belhaj** and S. Dadouch	**Dr. Mohamed Belhaj**, ONERA, University of Toulouse, **France**
18:00 - 18:15	Plasma surface treatment of aluminium sheets **P. I.Kovács**, M. Berczeli and Z. Weltsch	**Mr. Péter Kovács**, John von Neumann University, **Hungary**

17 April 2024		
SICT 2024 / Tribology 2024 Joint Session I. C: **Surface Engineering, Coatings and Tribology**		
Conference Room: Salon Schönbrunn 1		
Session's Chairs: **Prof. Auezhan Amanov, Tampere University, Finland** **Prof. Yonggang Meng, Tsinghua University, China** **Prof. Rafal Reizer, University of Rzeszow, Poland**		
14:00 - 14:30	The Astonishing Diversity in the World of 2D Materials - Lessons learned from MXenes and Transition Metal Carbo Chalcogenides (TMCCs) for Solid Lubrication **C. Gachot**	**Prof. Carsten Gachot,** Vienna University of Technology, **Austria**
14:30 - 14:45	The effect of ZrO2 nanoparticles reinforcement and heat treatment on tribological properties of electroless Ni-P coatings with high P content **E. Baroni**, G. Pedrizzetti, G. Pulci, F. Marra and M. Merlin	**Mr. Enrico Baroni**, University of Ferrara, **Italy**
14:45 - 15:00	A Comparative Study on the Effect of Roughness Parameters on the Adhesion and Tribological Properties of PEEK Coatings Deposited on Mild Steel Substrates **A.A. Seenath** and A.S. Mohammed	**Mr. Amal Ameen Seenath**, King Fahd University of Petroleum & Minerals, **Saudi Arabia**
15:00 - 15:15	A study of Ti-doped DLC coating applicable to gears for chassis parts to reduce friction and improve durability **H.S. Shin** and Y.K. Kim	**Mr. Heesup Shin**, Hyundai Motor Company, **Rep. of Korea**
15:15 - 15:30	Experimental study and modeling of tribo-chemical processes in transfer layer during friction in W-C:H coatings in various environments **F. Lofaj**, R. Bureš, M. Kabátová, H. Tanaka and Y. Sawae	**Dr. Frantisek Lofaj**, Institute of Materials Research of SAS, **Slovakia**
15:30 - 15:45	Residual stress development and thermo-elasto-plastic deformation in brake discs **Y. Kim** and M-G. Lee	**Mr. Youngjae Kim**, Hyundai Motor Company, **Rep. of Korea**
15:45 - 16:00	Influence of Coating on the Wear Performance of Wood Cutting Tools V.M. Nanshie, A. Aidibe, M. Heidari and **M. Jahazi**	**Prof. Mohammad Jahazi,** École de technologie supérieure (ÉTS), **Canada**
16:00 - 16:30	**Coffee Break / Posters Session**	
16:30 - 16:45	Compositionally modulated Ni-W multilayers to alleviate the residual stresses in coatings for superior wear resistance **N.P. Wasekar**, L. Bathini and MJNV Prasad	**Dr. Nitin Wasekar**, International Advanced Research Centre for Powder Metallurgy and New Materials, **India.**
16:45 - 17:00	Tailored Coatings for Enhanced Long-Term Tribological Performance of Journal Bearings in Accelerator-Driven Nuclear Reactors **E.Serag**, B. Caers, E. Haye, P. Schuurmans and S. Lucas	**Mr. Essam Serag**, University of Namur, **Belgium**

18 April 2024		
SICT 2024 Session II. A: **Surface and coatings Characterization / Properties** **Multifunctional composite and hybrid coatings**		
Conference Room: Salon Schönbrunn 2 / 3		
Session's Chairs: **Prof. Alberto Brambilla, Politecnico di Milano, Italy** **Prof. Stanko Brankovic, University of Houston, USA**		
09:00 - 09:30	Exploiting the natural instability in thin and flexible dielectric solid films for sensing and photonic applications **M. Bollani**	**Dr. Monica Bollani**, Photonic and Nanotechnology Institute, **Italy**
09:30 - 09:45	Role of Critical Coupling in planar optical nanodevices **E. Popov**	**Prof. Evgeni Popov**, Aix Marseille University, **France**
09:45 - 10:00	Optimization of Surface Reactivity of Diamond Particles by Atomic Layer Deposition of Alumina Coating **K. Shibuta**, H. Ishida, M. Groner andJ. Gauspohl	**Mr. Kaoru Shibuta**, SEKISUI CHEMICAL CO., LTD., **Japan**
10:00 - 10:15	Effect of Particles Dispersion Degree on Microstructure of Composite Plating for Ni-Magetite **T. Mori**, W. Murata, K. Nakamura, K. Kitamura, Y. Tanaka, K Fukui, A. Fujita and M. Takagi	**Prof. Takamasa Mori**, Hosei University, **Japan**
10:15 - 10:30	Effect of thermal treatment with different temperature-duration schedules on mechanical and corrosion properties of Ni-P nanocomposite coatings reinforced with ZrO2 particles **G. Pedrizzetti**, E. Baroni, M. Merlin, G. Pulci and F. Marra	**Ms. Giulia Pedrizzetti**, "La Sapienza" University of Rome, **Italy**
10:30 - 11:00	**Coffee Break / Posters Session**	
Session's Chairs: **Dr. Monica Bollani, Photonic and Nanotechnology Institute, Italy** **Prof. Stanko Brankovic, University of Houston, USA**		
11:00 - 11:30	Templating effects of graphene in stabilizing ultrathin compounds on metals **A.Brambilla**, A. Picone, M. Capra, S. Fiori, D. Dagur, G. Vinai, G. Panaccione and F. Ciccacci	**Prof. Alberto Brambilla**, Politecnico di Milano, **Italy**
11:30 – 11:45	Ultralight and robust current collectors for high-energy and high-safety lithium-ion batteries **S. He** and P. Gao	**Dr. Suihua He**, The Hong Kong University of Science and Technology, **China**
11:45 - 12:00	Growth behaviour of energy-efficient protective black PEO coatings on additively manufactured AlMgSi alloys **M.A. Iqbal**, Miguel Collado Vian, Raul Arrabal, Endzhe Matykina, Marta Mohedano	**Dr. Muhammad Ahsan Iqbal**, Complutense University Madrid, **Spain**
12:00 - 14:00	**Lunch Break – Restaurant**	
Group Photo at 13:45		

SICT 2024 / Plasma Tech 2024 Joint session II. B:
Bio-interfaces, Biomedical / Bioactive surfaces and coatings
Plasma applications for biology, medicine, and agriculture

Session's Chairs:
Prof. Holger Kersten, University Kiel, Germany
Prof. Rui Silva, University of Aveiro, Portugal
Prof. Sungmo Moon,Korea Institute of Materials Science, Rep. of Korea

14:00 - 14:30	Rapid inactivation of viruses in water by plasma treatments G. Primc, M. Zver, A. Filipič, D. Dobnik and **M. Mozetič**	**Prof. Miran Mozetic,** Jozef Stefan Institute, **Slovenia**
14:30 - 14:45	Well-defined poly(2-isopropenyl-2-oxazoline) brushes provide enhanced biocompatibility and versatility in surface functionalization M. Singh, L. Poláková, A. de los Santos Pereira, O. Pop-Georgievski, J. Svoboda, T. Riedel, S. Gupta, Z. Sedláková, V. Raus and **R. Poreba**	**Dr. Rafal Poreba**, Institute of Macromolecular Chemistry, **Czech Rep.**
14:45 - 15:00	Microstructure and Properties of Micro-Arc Oxidized Coatings Produced on Plastically Deformed Titanium **A. Trelka-Druzic**, F. Muhaffel, A. Jarzębska, G. Cempura, M. Kulczyk, H. Cimenoglu and Ł. Maj	**Mrs. Anna Trelka-Druzic**, Institute of Metallurgy and Materials Science, **Poland**
15:00 - 15:15	Atmospheric cold plasma technology advancements: enabling cost-effective, standardized and enhanced biomolecule immobilization for biosensing applications **L. Dankers**, K. Leirs, B. Nisol and J. Lammertyn	**Ms. Lieze Dankers**, KU Leuven, **Belgium**
15:15 - 15:30	Plasma Activated Media for Prostate Cancer Treatment **M. Moreau**, I. Ghzaiel, K. Charlet, S. Menecier, F. Perisse, M. Sleiman and M. Sarakha	**Mr. Maxime Moreau,** Clermont Auvergne University, **France**
15:30 - 15:45	Fundamentals of Plasma Disinfection for the Inactivation of Viruses **M. Shaban**, A. P. Weber and N. Merkert	**Dr. Masoom Shaban**, Clausthal University of Technology, **Germany**
15:45 - 16:00	Cold Plasma Induced Enhancement of Recombinant Thaumatin Bioproduction **N. Parsekar**, S. Akkermans, D. Kozak and J. F. M. Van Impe	**Ms. Nivedita Parsekar**, KU Leuven, **Belgium**
16:00 - 16:15	In vitro disinfection of MRSA biofilms with cold atmospheric plasma. Potential approach for hospital-acquired infection management **M. Lunder**, S. Dahle and R. Fink	**Mrs. Manca Lunder**, University of Ljubljana, **Slovenia**
16:00 - 16:30	**Coffee Break / Posters Session**	

Conference Dinner

19:30 – Bristol Lounge

18 April 2024		
SICT 2024 / Plasma Tech 2024 Joint Session II. C: Plasma fundamentals / Modelling / Atomic and Molecular Processes Plasma Processing / Materials Interactions / Coatings		
Conference Room: Salon Bristol		
Session's Chairs: Prof. Yasunori Tanaka, Kanazawa University, Japan Dr. Nicolas Naudé, LAPLACE, University de Toulouse, France		
08:30 - 09:00	EUV Generated Hydrogen Plasmas T. Piskin and **M.J. Kushner**	**Prof. Mark Kushner**, University of Michigan, **USA**
09:00 - 09:30	Renewable electricity driven chemistry for energy conversion and storage: Novel pathways provided by plasma enhanced chemistry **R. van de Sanden**	**Prof. Richard van de Sanden**, Eindhoven University of Technology, **The Netherlands**
09:30 - 09:45	Multi-Scale Model for High Aspect Ratio TiN Etching in a Cl2/Ar Inductively Coupled Plasma **L. Filipovic** and T. Reiter	**Prof. Lado Filipovic**, Institute for Microelectronics, TU Wien, **Austria**
09:45 - 10:00	Simple Plasma-triggered Reduction & Exfoliation of Graphene Oxide and Fabrication of 2D and 3D rGO Structures **R. Krumpolec**, F. Zelenák, J. Jurmanová, M. Stupavská, M. Pazderka, Z. Moravec and M. Černák	**Dr. Richard Krumpolec**, Masaryk University, **Czech Rep.**
10:00 - 10:15	Non-Thermal Plasma Synthesis of Imine Macrocycles **P. Roszkowska**, A. Scholes, J.L. Walsh, T.L. Easun and A.G. Slater	**Ms. Patrycja Roszkowska**, University of Liverpool, **UK**
10:15 - 10:30	Plasma Diagnostics for Ion Energy and Flux measurements in Etching and Deposition processes A. Verma and T. Gilmore	**Mr. Thomas Gilmore**, Impedans Ltd, **Ireland**
10:30 - 11:00	**Coffee Break / Posters Session**	
Session's Chairs: Prof. Yasunori Tanaka, Kanazawa University, Japan Prof. Richard van de Sanden, Eindhoven University of Technology, The Netherlands Dr. Nicolas Naudé, LAPLACE, University de Toulouse, France		
11:00 - 11:15	The effect of plasma parameters on the surface treatment of electrocatalysts **H. Li**, C. Schulze and J. Benedikt	**Mr. He Li**, Kiel University, Kiel, **Germany**
11:15 - 11:30	Surface Functionalization of Industrial Materials via PECVD and its Influence on Oxygen Nucleation **J. Heinrich**, K. Schwarzenberger, X. Yang and K. Eckert	**Mr. Julian Heinrich**,, Helmholtz-Zentrum Dresden-Rossendorf, **Germany**
11:30 - 11:45	Atmospheric Pressure cold Plasma processing of textiles – applications in functional finishings **S.S. Palaskar**	**Dr. Shital S. Palaskar** The Bombay Textile Research Association **India**
11:45 - 12:00	Enhancing adhesion of recycled and non-recycled EVA via atmospheric pressure plasma treatments **C. Ruzafa-Silvestre**, V.M. Serrano-Martínez, M.D. Romero-Sánchez and E.Orgilés-Calpena	**Mr. Carlos Ruzafa Silvestre**, INESCOP, **Spain**
12:00 - 12:15	Enhancing Cotton Fabrics Dyeability through Low-Pressure Plasma Activation Surface Treatment V. M. Serrano Martínez, C. Ruzafa Silvestre, M. D. Romero Sánchez and E. Orgilés Calpena	**Mr. Victor M. Serrano Martínez**, INESCOP, **Spain**
12:15 - 12:30	Industrially Applicable Atmospheric-Pressure Plasma Treatment of Nonwovens, Cotton and Electrospun Nanofibres for the Enhancement of Their Performance Properties in Various Areas of Applications **D. Kováčik**, O. Galmiz, J. Kelar, L. Zahedi, S. Ben Hamida, V. Medvecká, R. Krumpolec, L. Mynářová and M. Černák	**Prof. Dusan Kovacik,** Masaryk University, **Czech Rep**
12:00 - 14:00	**Lunch Break - Restaurant**	
Group Photo at 13:45		

Session's Chairs: **Prof. Catherine Batiot-Dupeyrat, University of Poitiers, France** **Prof. Wen-Bin Jian, National Yang Ming Chiao Tung University, Taiwan** **Prof Claudia Riccardi, University Milano-Bicocca, Italy**		
14:00 - 14:30	High-Throughput Synthesis of Nanomaterials by Spatial-Temporal Control of Modulated Induction Thermal Plasma Fields and its Optimization by Machine Learning **Y. Tanaka**	**Prof. Yasunori Tanaka**, Kanazawa University, **Japan**
14:30 - 15:00	Diffuse Dielectric Barrier Discharges at atmospheric pressure: Technical and Scientific locks for thin film deposition **N. Naudé**, A. Belinger, H. Caquineau, S. Dap and F. Fanelli	**Dr. Nicolas Naudé**, LAPLACE, University de Toulouse, **France**
15:00 - 15:15	Plasma-Assisted Low-Temperature Curing of Polysilazane Coating on Aluminum Substrate **P. Ghourchi Beigi**, R. Krumpolec, L. Zahedi, M.Stupavská and D. Kováčik	**Mr. Pedram Ghourchi Beigi**, Masaryk University, **Czech Rep.**
15:15 - 15:30	An engineering and a chemical approach to overcome inhibition of polymerization during open air atmospheric pressure plasma deposition of poly(ethylene oxide) coatings **T. Dekoster**, R. Vos, W. Van Roy, B. Nisol, K. Jans and A. Delabie	**Mr. Tijs Dekoster**, KU Leuven, Leuven, **Belgium**
15:30 - 15:45	Study of adhesion promoter coatings, obtained by atmospheric pressure plasma, on granulous substrates **I. Bacquet**, C. Guyon, M. Yonger and M. Tatoulian	**Mrs. Irène Bacquet,** Institut de Recherche de Chimie Paris/ Saint-Gobain Recherche Paris, **France**
15:45 - 16:00	Effect of Positive Voltage Pulses in HiPIMS on the Ti-PEEK Interface Formation **L. Cvrček**, V. Nehasil and M.Buřil	**Dr. Ladislav Cvrček**, Czech Technical University in Prague, **Czech Rep**
16:00 - 16:30	**Coffee Break / Posters Session**	
Session's Chairs: **Prof. Jochen Schein, University of Federal Armed Forces Munich, Germany** **Prof. Wen-Bin Jian, National Yang Ming Chiao Tung University, Taiwan** **Prof. Dusan Kovacik, Masaryk University, Czech Rep.**		
16:30 - 16:45	Synthesis of carbon micro- and nanostructures by means of DC PACVD **M. Schachinger**, F. A. Delfin, B. Fickl, B. C. Bayer, C. Forsich and D. Heim	**Mr. Manuel Schachinger**, University of Applied Sciences Upper Austria, **Austria**
16:45 - 17:00	Atmospheric-pressure Plasma Enhanced Chemical Vapor Deposition of size agents on fiberglass **M. Troia**, A. Schulz, M. Walker, M. Haag, D. Ben Salem, P. Holste, A. Knospe, C. Dobslaw and B. Glocker	**Dr. Mariagrazia Troia**, University of Stuttgart, **Germany**
17:00 - 17:15	Non-thermal plasma in nanoscale: application potential in the preparation of silicon quantum dots and tailoring of their surface chemistry using plasma-activated liquids. **P Galář**, F. Matějka, J. Kopenec and K. Kůsová	**Dr. Pavel Galář**, Charles University- Prague, **Czech Rep.**
17:15 - 17:30	Adaptation of non-thermal plasma system to overcome size limitations of sythetized silicon nanoparticles **J. Kopenec**, F. Matějka, P. Galář and K. Kůsová	**Mr. Jakub Kopenec**, Charles University- Prague, **Czech Rep.**
17:30 - 17:45	Plasma Jet Sputtering as an Efficient Tool for the Preparation of Transition Metal Oxide Catalysts Supported on Stainless Steel Meshes **P. Topka**, K. Jirátová, M. Čada, I. Naiko, A. Ostapenko, J. Balabánová, M. Koštejn, J. Maixner, Z. Hubička and F. Kovanda	**Dr. Pavel Topka**, Institute of Chemical Process Fundamentals, **Czech Republic**
17:45 - 18:00	General and Scalabe Synthesis of High-entropy Alloy Nanoparticles by Thermal Plasma **K.S. Kim**, M. Couillard, Z. Tang, H. Shin, D. Poitras, C. Cheng, O. Naboka, D. Ruth, M. Plunkett, L. Chen, L. Gaburici, T. Lacelle, M. Nganbe and Y. Zou	**Dr. Keun Su Kim**, National Research Council Canada, **Canada**
18:00 - 18:15	Synthesis of Simulated Radioactive Post-Detonation Particles by DC Plasma **G. Cota-Sanchez**, M. Martinez, A. Chaudhuri, M. Totland, J. Garcia-Alonso and N. Lee	**Dr. German Cota-Sanchez**, Canadian Nuclear Laboratories, **Canada**

18:15 - 18:30	Investigation of ionized metal flux fraction of magnetron sputtering at industrial conditions **P. Vašina**, J.Hnilica P. Klein, S. Debnárová, V. Sochora, M. Učík, J. Klusoň, M. Jílek and A. Lümkemann	**Prof. Petr Vasina**, Masaryk University, **Czech Rep.**
18:30 - 18:45	Active turbulence in a 2D complex plasma with Janus particles **V. Nosenko**	**Dr. Volodymyr Nosenko**, German Aerospace Center, **Germany**
18:45 - 19:00	Plasma sprayed porous Raney Nickel cathodes for alkaline water electrolysis **M. Wetegrove**, U. Lindemann and A. Kruth	**Dr. Marcel Wetegrove**, Leibniz Institute for Plasma Science and Technology, **Germany**

Conference Dinner
19:30 – Bristol Lounge

18 April 2024		
Tribology 2024 Session II. D: **Coatings and Surfaces Corrosion / Tribological Properties /** **Physics or Chemistry of Tribo-Surfaces/ Nanotribology**		
Conference Room: Salon Schönbrunn 1		
Session’s Chairs: **Prof. Carsten Gachot, Vienna University of Technology, Austria** **Prof. Pawel Pawlus, Rzeszow University of Technology, Poland** **Prof. Giovanni Straffelini, University of Trento, Italy**		
09:30 - 10:00	Solving tribological challenges in the era of Digital Transformation and supporting Circular Economy as well as Energy and Mobility Transition **E. Badisch**	**Dr. Ewald Badisch**, AC2T Research GmbH, **Austria**
10:00 - 10:30	Controlling surface tribological properties reduces the environmental impact of engineering technologies. **G. Carbone**	**Prof. Giuseppe Carbone**, Politecnico di Bari, **Italy**
10:30 - 11:00	**Coffee Break / Posters Session**	
Session’s Chairs: **Dr. Ewald Badisch, AC2T Research GmbH, Austria** **Prof. Pawel Pawlus, Rzeszow University of Technology, Poland** **Prof. Giovanni Straffelini, University of Trento, Italy**		
11:00 - 11:30	Application of Surface Modification Technology for Improving the Tribological Properties of Additively Manufactured Materials **A.Amanov**	**Prof. Auezhan Amanov**, Tampere University, **Finland**
11:30 - 11:45	Friction-assisted Selective Electrodeposition of FeCoNi Alloy Film on Pre-wear Surface of Bearing Steels Y. Song, C. Liu and **Y. Meng**	**Prof. Yonggang Meng**, Tsinghua University, **China**
11:45 - 12:00	Investigation of the tribological properties and tribo-layers of Graphene in Cu metal matrix composites. **Y. Xu**, P. Yao and H. Zhou	**Mr. Yuxuan Xu**, Central South University, **China**
12:00 - 12:15	Brake emissions and performance of a WC-coated disc sliding against a NAO friction material **S. Candeo**, A.P. Nogueira and G. Straffelini	**Mr. Stefano Candeo**, University of Trento, **Italy**
12:00 - 14:00	**Lunch Break - Restaurant**	
Group Photo at 13:45		
Session’s Chairs: **Prof. Giuseppe Carbone, Politecnico di Bari, Italy** **Prof. Auezhan Amanov, Tampere University, Finland**		
14:00 - 14:15	Effect of laser texturing on the frictional resistance in reciprocating motion at elevated temperature S. Wos, W. Koszela, A. Dzierwa, J. Sep and **P. Pawlus**	**Prof. Pawel Pawlus**, Rzeszow University of Technology, **Poland**
14:15 - 14:30	In-depth analysis of the tribological response of PHBV and its blends with PLA via scratch testing **P. M. Martínez-Rubio**, M. D. Avilés, L. Mínguez, R. Pamies, and F. J. Carrión-Vilches	**Mr. Pablo M. Martínez-Rubio**, Polytechnic University Cartagena, **Spain**
14:30 - 14:45	The Influence of Retained Austenite and Microstructure on the Abrasive Wear Resistance of Bainitic Steels **C.S. Brown**, J.G. Speer and E. De Moor	**Ms. Caseyq Brown**, Colorado School of Mines, **USA**
14:45 - 15:00	Microstructural evolution of scuffing in self-mated steels measured in-situ during sliding **S. Berkebile**, C. Lorenzo Martin, D. Bachnacki, F. Ahmed Koly, N. Murthy, S. Walck, J.-S. Park, P. Kenesei, D. Burris, O. Ajayi, S. Liu, A. Butler-Christodoulou, A. Bhattacharjee, Q. J. Wang and B. Gould	**Dr. Stephen Berkebile**, DEVCOM Army Research Laboratory, **USA**
15:00 - 15:15	Measuring Bearing Loads – Practical Implementation of Impedance Measurement in a Machine Tool Spindle **S. Puchtler**, M. Fett, G. Martin and E. Kirchner	**Mr. Steffen Puchtler**, Technical University of Darmstadt, **Germany**

15:15 - 15:30	BaTiO3 /UHMWPE Composites for Enhanced Performance in Load-Bearing Biomedical Implants **D. Havaldar**, Z. Starý, L. Cvrček, J. Walter, Z. Jeníková and K. Pawar	**Ms. Darshana Havaldar**, Czech Technical University in Prague, **Czech Rep.**
15;30 - 15:45	Role of terminations on the nano-tribological properties of Ti-based MXene layers studied by DFT simulations **E. Marquis**, F.Benini, A.Rosenkranz and M.C. Righi	**Mr. Edoardo Marquis**, University of Bologna, **Italy**
15;45 - 16: 00	The Influence of Pin Inclination on Frictional Behavior in Pin-on-Disc Sliding and Its Implications for Test Reliability **H. Yue**, J. Schneider and P. Gumbsch	**Dr. Hongzhi Yue, Karlsruhe,** Institute of Technology, **Germany**
16:00 - 16:30	**Coffee Break / Posters Session**	
16:30 - 16:45	A novel MTES/polysilazane hybrid polymer for corrosion protection of stainless steel substrate **P. N. Moghaddam**, M. Parchovianský, A. Duran, E. Merino, Y. Castro, I. Parchovianská and A. Pakseresht	**Mrs. Parisa N. moghaddam**, Alexander Dubček University of Trenčín, **Slovakia**
16:45 - 17:00	Exploring the Relationship Between Surface Treatment Processes and Tensile Strength in Steel Bonds P. I. Kovács, B. Körömi, Z. Weltsch and **M. Berczeli**	**Dr. Miklós Berczeli**, John von Neumann University, **Hungary**
17:00 - 17:15	Li-ION battery cell bonding technology **F. Tajti**, M. Berczeli and Z. Weltsch	**Mr. Ferenc Tajti**, John von Neumann University, **Hungary**

Conference Dinner
19:30 – Bristol Lounge

19 April 2024

Tribology 2024 Session III. A:
Lubricants and hydrodynamic lubrication / Biotribology

Conference Room: Salon Bristol

Session's Chairs:
Prof. Yonggang Meng, Tsinghua University, China
Prof. Giovanni Straffelini, University of Trento, Italy

09:15 - 09:45	Recent Advances and Current Challenges in Developing Predictive Models and Design Tools in Tribology **D. Dini**	**Prof. Daniele Dini**, Imperial College London, **UK**
09:45 - 10:00	Tribological behaviours of additively manufactured CoCrMo in bovine calf serum for human joint replacement: understanding the role of process parameters and porosity **Q. Shi**, M.S. Abd Aziz, T. Reddyhoff and C. Myant	**Ms. Qingyue Shi**, Imperial College London, **UK**
10:00 - 10:30	**Coffee Break / Posters Session**	
10:30 - 11:00	PVD nitrides to be used for increased life time of tools and components **Paul H. Mayrhofer**	**Prof. Paul H. Mayrhofer**, TU Wien, **Austria**
11:00 - 11:15	Insight into the fundamental behaviour of Organic Friction Modifiers doped with small polar molecules **I. Kicior**, T. Poręba, E. A. Willneff, J. Frey, V. Honkimäki, A. Morina, P. Dowding and S. L. M. Schroeder	**Ms. Inga Kicior**, European Synchrotron Radiation Facility, Grenoble, **France**
11:15 - 11:30	Ecolubricants based on vegetable oils with ionic liquid **M.D. Avilés,** P. Mostaza-Ucedo, T. Caparrós and F.J. Carrion-Vilches	**Dr. María D. Avilés González,** Polytechnic university of Cartagena, **Spain**
11:30 - 11:45	Optimizing Tribological Performance: Exploring the Role of Surface Texture and Lubricants Composition **M.S. Abd Aziz**, T. Reddyhoff, C. Tadokoro	**Mr. Mohd Syafiq Abd Aziz**, Imperial College London, **UK**
11:45 - 12:00	A bearing test stand to represent E-VTOL air-screw loads in vertical and forwards flight. **R. T. Wragge-Morley**, G. C. Barnaby, J. M. Yon and P. H. Mellor	**Dr. Robert Wragge-Morley**, University of Bristol, **UK**
12:00 - 12:15	Superlubricity of carbon films via regulated interface **B. Zhang**	**Prof. Bin Zhang**, Lanzhou Institute of Chemical Physics, **China**

19 April 2024

SICT 2024 / Plasma Tech 2024 Session III. B: Coatings for Energy and Environmental Applications Plasma application in Energy and environment

Conference Room: Salon Schönbrunn

Session's Chairs:
Prof. Lado Filipovic, TU Wien, Austria
Prof. Mark Kushner, University of Michigan, USA
Prof. Jochen Schein, University of Federal Armed Forces Munich, Germany

08:30 - 09:00	Non-thermal plasma: an efficient technology for regeneration of coked zeolite **C. Batiot Dupeyrat**	**Prof. Catherine Batiot-Dupeyrat**, University of Poitiers, **France**
09:00 - 09:30	Preparation of Nanoparticulate WO3/MoO3 Films for Making Electro-chromic and Energy-Storage Devices **W-B. Jian**, C-C W;ei, P-H. Lin, T-H. Wu, Jun-Wei Huang	**Prof. Wen-Bin Jian**, National Yang Ming Chiao Tung University, **Taiwan**
09:30 - 10:00	Room Temperature Synthesis of Hydrogen Permeation Barrier for Storage and Transportation Application **S. R. Brankovic**, N. Amiri and I. Rabbi	**Prof. Stanko Brankovic**, University of Houston, **USA**
10:00 - 10:30	**Coffee Break**	
10:30 - 10:45	Film-Based Repair System for Offshore Wind Tower Coatings **T. Marquardt**, A.W. Momber, S. Buchbach, M. Irmer, M. Beitmann and B. Koeller	**Mr. Tom Marquardt**, Muehlhan Holding GmbH, **Germany**
10:45 - 11:00	Development of a 100 kW Steam Plasma Torch (SPT) for Waste Remediation and Clean Hydrogen Production **H.R. Yousefi**, B. Glocker, S. Pauly and H. Koch	**Dr. Hamid Reza Yousefi**, PlasmaAir AG, **Germany**
11:00 - 11:15	Atomic Scale Loading of Palladium on Oxygen-Deficient Cerium Oxide for Electrocatalytic Applications A.B. Yousaf and **P. Kasak**	**Dr. Peter Kasak**, Qatar University, **Qatar**
11:15 - 11:30	Novel deposition technique of α-Al2O3-based coating as a hydrogen permeation barrier **N. Laadel**, M. El Mansori, N. Frederich and S. Marlin	**Mr. Nour-Eddine Laadel**, Arts et Métier Institute of Technology, France
11:30 - 11:45	Reactive sputtering of ceramic hydrogen barrier coatings **A. Zirbel**, L. Haus, S. Hübner, M. Müller, J. Riedel and S. Ulrich	**Mrs. Anne Zirbel**, Robert Bosch Manufacturing Solutions GmbH, **Germany**
11:45 - 12:00	Influence of operating parameters of nanosecond pulsed dielectric dis-charge on the CH4 reforming with CO2 **T.V. Reddy Thurpu**, E. Fourre S. Rossignol and C. Batiot Dupeyrat	**Mr. Teja Vardhan Reddy Thurpu**, University of Poitiers, **France**
12:00 - 12:15	Original liquid DBD plasma reactor coupled with membrane filtration N. Mougel, **E. Fourré** and B. Teychené	**Dr. Elodie Fourré**, University of Poitiers, **France**
12:15 - 12:30	Hydrogen Production from NH3 in a Ferroelectric Packed-Bed Plasma Reactor **M. Ruiz-Martín**, S. Marín-Meana, A. Megías-Sánchez, M. Oliva-Ramírez, J. Cotrino, A. R. González-Elipe and A. Gómez-Ramírez	**Mr. Mateo Ruiz Martín**, University of Seville, **Spain**

SICT 2024 / Plasma Tech 2024 / Tribology 2024 Joint Conferences Posters

17 and 18 April 2024 Sessions

N.	Poster Title	Author, Affiliation, Country
1.	Design and synthesis of advanced nanostructured coatings with hydrophobic/ icephobic properties for aviation **Z. Sideratou**, D. Tsiourvas, F. Katsaros, K. SpyroU, A. Sapalidis, S. Papageorgiou and P. Booker	**Dr. Zili Sideratou**, NCSR “Demokritos”, **Greece**
2.	Methodological study of the deposition parameters of aminopropyl silsesquioxane on the surface of glass vials using aqueous solution **T. Pastore**, G. Trevisi, F. Casoli , L. Savio, M. Di Maro, G. Gautier, M. G. Faga, D. Costa, M. Poncini and D. Faverzani	**Ms. Tiziana Pastore**, University of Parma, **Italy**
3.	Multilayered CVD coated tools with texturized Al2O3 layer for machining of high strength special steels A. Pinto, J. Ferreira, **F.J. Oliveira**, A.V. Girão, C.M. Fernandes and D. Figueiredo	**Dr. Filipe J. Oliveira**, University of Aveiro, **Portugal**
4.	Comparative investigation of the biodegradable control properties of post- and simply PEO coated Mg alloy **Z. Ur Rehman** and B. Heun Koo	**Dr. Zeeshan Ur Rehman**, Changwon National University, **Rep. of Korea**
5.	The high-temperature stability and mechanical properties of multi-component TiTaZrHfxN coatings prepared by reactive DC magnetron sputtering and High target utilization sputtering **L. Kvetková**, P. Hviščová, F. Lofaj, M. Mikula and T. Roch	**Mrs. Lenka Kvetkova**, Institute of Materials Research SAS, **Slovakia**
6.	Nanoarchitectures with Cu-Ag embedded clusters for enhanced localized surface plasmon resonance detection **S. Kumar**, H. Maskova, L. Doudová, P. Pleskunov, A. Kuzminova, P. Curda, L. Hejduk, J. Sterba, O. Kylián, R.O.M. Rego and V. Stranak	**Mr. Sanjay Kumar**, University of South Bohemia, **Czech Rep.**
7.	Tribogical properties of DLC deposited by PE-CVD under high Vacuum **B.R. Cho**,T-H. Jang,T-G. Kim,I. Heo, B. Choe and J. H. Kim	**Mr. Bo Ram Cho**, Korea Institute of Industrial Technology, **Rep. of Korea**
8.	Structural and mechanical properties of laminate-type thin film SWCNT/ SiOXNY composites **E. Shmagina**, O. Volobujeva, M. Antonov and S. Bereznev	**Ms. Elizaveta Shmagina**, Tallinn University of Technology, **Estonia**
9.	Mechanical endurance improvement of the coated nanosurface by laser Y.N. Jung and **M. Yung Jeong**	**Prof. MyungYung Jeong**, Pusan National University, **Rep. of Korea**
10.	Hybrid SLM/DED Additive Manufacturing of Ti6Al4V Alloy-Optimization of Process Parameters and Heat Treatment **A.Kocijan**	**Dr. Aleksandra Kocijan**, Institute of Metals and Technology, **Slovenia**
11.	Study of 2D Materials by Time-of-Flight Spectroscopy **I. Konvalina**, E. Materna Mikmeková, L. Průcha, J. Piňos, A. Paták, M. Zouhar and I. Mül-lerová	**Dr. Ivo Konvalina**, Institute of Scientific Instruments of the Czech Academy of Sciences, **Czech Rep.**
12.	Dynamics of compound droplet with dual viscosities on obstacles for center-assisted retraction **S. Yun**	**Prof. Sungchan Yun**, Korea National University of Transportation, **Rep. of Korea**
13.	On the use of a plasma jet for atomic oxygen production: Source characterization and application in cultural heritage **M. Poupouzas**, A. Nikiforov, R. Morent and A. Sobota	**Mr. Michail Poupouzas**, Eindhoven University of Technology, **The Netherlands**
14.	Slot-die coating of cellulose nanocrystals for improved barrier properties of paper **V. Kokol**	**Mr. Vanja Kokol**, University of Maribor, **Slovenia**
15.	Atmospheric pressure glow discharge for the determination of hydride-forming elements **K. Gręda**, J. Kratzer, M. Svoboda, N. Vlčková, J. Dědina and P. Pohl	**Dr. Krzysztof Greda**, Wroclaw University of Science and Technology, **Poland**
16.	Instabilities in O2 Capacitively Coupled Radio-Frequency Plasmas **M. Myrzaly**, R. Masheyeva, C. Tian, M.Vass, L.Y. Luo, K. Dzhumagulova, P. Hart-mann, J. Schulze and Z. Donkó	**Mr. Murat Myrzaly**, Satbayev University, **Kazakhstan**
17.	Study of the surface and effect of cold plasma treatment on the adhesion of Polyphenylene Sulfide **M.A. Martinez** and J. Abenojar	**Dr. Miguel Angel Martinez**,University Carlos III of Madrid, **Spain**

18.	Hierarchically Nanostructured metal oxide thin films by plasma **C. Riccardi** and H.E. Roman,	**Prof Claudia Riccardi,** University Milano-Bicocca, **Italy**
19.	Fabrication of Atmospheric Plasma-reduced rGO-based Nanocomposites and Its Applications **F. Zelenák**, R. Krumpolec , J. Jurmanová, M.Stupavská, M. Kováčová, P. Neilinger, Z. Moravec, M. Grajcar and M. Černák	**Mr. František Zelenák**, Masaryk University, **Czech Rep.**
20.	Effect of molybdenum concentration and deposition temperature on the structure and tribological properties of the diamond like-carbon films. **H. Zhairabany**, A. Sarakovskis, E. Gnecco, E. Vanags and L. Marcinauskas	**Mr. Hassan Zhairabany**, Kaunas University of Technology, **Lithuania**
21.	Towards green tribology: water versus oil lubrication of laser-textured Ti6Al4V alloy upon addition of MoS2 nanotubes **M. Conradi**, B. Podgornik, A. Kocijan, M. Remškar and D. Klobčar	**Dr. Marjetka Conradi**, Institute of metals and technology, **Slovenia**
22.	The effect of shape and size of nanoceria on the antimicrobial properties of nanocoatings Z. Sideratou, A. Papavasiliou, K.M. Lyra, D. Tsiourvas, L. Hernández Ruiz, T. Oroz Mateo and **F.K. Katsaros**	**Dr. Fotios Katsaros**, NCSR “Demokritos”, **Greece**
23.	Development of nanoporous nickel oxide materials as electrodes for supercapacitors **Y-M. Lu**, Y-C.Lin and L. Ting Yi	**Prof. Yang-Ming Lu**, National University of Tainan, **Taiwan**
24.	Saving Energy in Electrochemical Water Treatment Using BDD Electrodes Through Forced Fouling-Reactivation Cycles: Optimizing Frequency and Duty Cycle P. Brosler, **R. F. Silva**, J. Tedim and F. J. Oliveira	**Prof. Rui Silva**, University of Aveiro, **Portugal**
25.	Effect of plasma on cork particles to adsorb magnetite, and later, hexavalent chromium from water. **J. Abenojar**, S. López de Armentia, M.A. Martínez and J.C. del Real	**Dr. Juana Abenojar**, University Carlos III of Madrid, **Spain**
26.	Improvement of Hydrophilicity of Composite Membrane and its application for Desalination P. Rattana-arporn and **S. Poompradub**	**Dr. Sirilux Poompradub**, Chulalongkorn University- Bangkok, **Thailand**
27.	N2/Ar dual-gas microplasma device conducting first-in-human wound treatment clinical trial S-C. Pan, Y-H. Wei, M-H. Cheng and **J-D. Liao**	**Prof. Jiunn-Der Liao**, National Cheng Kung University- Tainan, **Taiwan**
28.	Dielectric barrier discharge air plasma as a sustainable process for bio-refinery development towards poly(3-hydroxybutyrate) production **C. Argeiti**, E. Stylianou, D. Ladakis, P.J Cullen and A. Koutinas	**Mrs. Chrysanthi Argeiti**, Agricultural University of Athens, **Greece**
29.	Biorefinery electrification by dielectric barrier discharge plasma of lignocellulo-sic biomass in a semi-pilot microbubble reactor **E. Georgiadou,** N. Giannakis, K. Filippi and A. Koutinas	**Ms. Evanthia Georgiadou**, Agricultural University of Athens, **Greece**
30.	Radiative transfer in air-H2O mixtures for LIBS applications: calculation of the Net Emission Coefficients and the Mean Absorption Coefficients **I. Bendida**, Y. Cressault, B. Liani and F. Valensi	**Mrs. Imane Bendida**, University of Paul Sabatier Toulouse , **France**
31.	Simultaneous production of hydrogen gas and carbon black nanoparticles using liquid phase plasma method **S.-C. Jung** and K.-H An	**Prof. Sang-Chul Jung**, Sunchon National University, **Rep. of Korea**
32.	Catalytic effects of the load-induced confinement within metallic interfaces on molecular dissociation **M. Vezzelli**, E. Marquis and M.C. Righi	**Mr. Matteo Vezzelli**, University of Bologna, **Italy**
33.	Ni-Al2O3 Cold Spray Coating: Effect of process parameters on Microstructure and Performance of the coating **R.A. Goriya**, A.A. Sorour and N. Ogunlakin	**Mr. Raihan A. Goriya**, King Fahd University of Petroleum & Minerals, Dhahran, **Saudi Arabia**
34.	Abrasion of Polyester Sportswear Materials: The Impact of Yarn Parameters and Aging G. Čubrić, **I.** Salopek Čubrić and **A. Petrov**	**Ms. Antonija Petrov**, University of Zagreb Faculty of Textile Technology, **Croatia**
35.	Effect of ceramic tribo-elements on tribological properties of rough steel surfaces A. Dzierwa and **R. Reizer**	**Prof. Rafal Reizer**, University of Rzeszow, **Poland**
36.	Multi-criteria optimization of tribological properties of slide-burnished 36NiCrMo16 steel **A. Dzierwa**, J. Sep and P. Pawlus	**Dr. Andrzej Dzierwa**, Rzeszow University of Technology, **Poland**

37.	Evaluation Method for Dynamic Damage Performance of Materials under High-Frequency Impact **M. Hua** and X. Sui	**Mr. Minqi Hua**, Lanzhou Huahui Instrument Technology Co., Ltd, **China**
38.	Protic ionic liquids as additives in water-based ecolubricants **P. Mostaza-Ucedo**, J. Arias, M.D. Avilés and F.J. Carrion-Vilches	**Ms. Paloma Mostaza Ucedo**, Universidad Politécnica de Cartagena, **Spain**
39.	Influence of Tribo-Films of Calcium Detergents on Micro-Pitting Behaviour of Steels **A. Tada**, D. Spaltmann, K. Tagawa and V. L. Popov	**Mr. Akira Tada**, Technical University of Berlin, **Germany**
40.	Effect of vacuum atomic oxygen irradiation on the tribological properties of fullerene-like carbon and MoS2 films **K. Gao**, B. Zhang and J. Zhang.	**Dr. Kaixiong Gao**, Lanzhou Institute of Chemical Physics, **China**

SICT 2024 / Plasma Tech 2024 / Tribology 2024 Joint Plenary Session

Plasma Chemical Surface Engineering

D. Hegemann [1,*], P. Navascués [1]
[1] Plasma & Coating, Empa - Swiss Federal Laboratories for Materials Science and Technology, St.Gallen, Switzerland

Abstract:

Low-pressure plasma technology presents a versatile and powerful surface engineering method. Plasma surface engineering covers different approaches from surface activation and etching to the deposition of uniform thin films or coatings comprising nanostructures. Using direct plasma exposure (DPE), the plasma interaction is assisted by energetic particles, mainly positively charged ions, depositing energy during ablation or deposition processes. While this interaction is often beneficial, it might also induce damaging of the sample or the growing film structure.

Therefore, different approaches such as remote plasma (afterglow, downstream) have been applied to lower the ion bombardment, however, also lowering the density of reactive species. We thus introduce a novel concept, named near-plasma chemistry (NPC), by placing a polymeric mesh inbetween plasma and sample, i.e. in the sheath region, avoiding secondary plasma ignition [1]. The plasma-chemical activation in the gas phase remains unaffected, important to generate the reactive species as governed by the specific energy input [2]. Due to the negative surface charging of the mesh, positive ions are effectively attracted. Hence, only fast neutrals as formed by charge exchange are able to pass and reach the sample besides all other neutral reactive species as depicted in Figure 1. Furthermore, fast electrons pass the mesh, supporting the plasma-surface interaction, while room temperature conditions are present.

The NPC configuration was applied to the plasma activation of poly (tetrafluorethylene) (PTFE) samples, demonstrating reduced hydrophobic recovery compared to DPE. A more defined plasma-chemical interaction avoiding damaging was inferred for NPC as studied by ATR-FTIR [1]. Moreover, the plasma deposition of SiOx films using hexamethyldisiloxane (HMDSO) followed by oxygen etching of residual hydrocarbons was investigated. The NPC configuration allowed a significantly enhanced porosity of the SiOx nanostructure up to about 25 vol% [1]. These findings are of great interest for diffusion control, gas selection, and as low-k dielectrics that can be deposited on sensitive substrate materials due to the mild NPC conditions operating at room temperature. Furthermore, similar conditions apply when samples with complex geometries such as textiles, nonwovens, scaffolds, membranes etc. are considered. Here, the plasma-chemical interaction can be used to improve the penetration depth of the plasma functionalization by diffusion of reactive species showing different surface reactivities [3].

Keywords: plasma-surface interaction, reactive species, plasma activation, plasma deposition, hydrophobic recovery, porosity, plasma penetration depth.

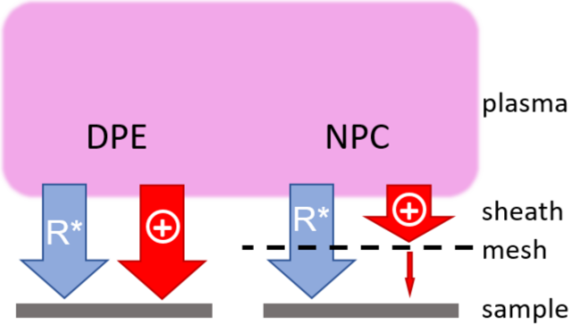


Figure 1: Figure illustrating the difference between direct plasma exposure (DPE) and near-plasma chemistry (NPC), whereby the mesh effectively hinders ions to reach the sample.

References:

1. Navascués, P., Schütz, U., Hanselmann, B., Hegemann, D. (2024), Near-Plasma Chemical Surface Engineering, *Nanomaterials*, In Press.
2. Hegemann, D. (2023), Plasma activation mechanisms governed by specific energy input: Potential and perspectives, *Plasma Process. Polym.* 20, e2300010.
3. Navascués, P., Buchtelová, M., Zajícková, L., Rupper, P., Hegemann, D. (2023), Polymerization mechanisms of hexamethyldisiloxane in low-pressure plasmas involving complex geometries, *Appl. Surf. Sci.*, 645, 158824.

Diagnostic of plasma processes for electric space propulsion

H. Kersten, T. Trottenberg, V. Schneider, A. Spethmann, R. Wimmer-Schweingruber
Institute for Experimental and Applied Physics, University Kiel, Kiel, Germany

Abstract:

Electric propulsion is becoming a standard drive technology for satellites and space probes. This way, much higher exhaust velocities are achieved than in case of chemical engines, where the effective exhaust velocity is roughly the thermal speed of the hot gas. Mostly, solar-electric power is used to ionize the propellant gas and electrostatically accelerate the ions.

Unfortunately, plasma based thrusters inherently suffer from a certain amount of neutral gas that unintentionally leaks through the open side of the discharge channel. The neutral gas interacts with the accelerated ions by means of collisions, in particular by charge-exchange collisions. The latter generate ions with low thermal velocities that form a secondary plasma surrounding the spacecraft (the primary plasma meaning the neutralized exhaust plume) [1,2]. The plasma surrounding the spacecraft attains, as all low-temperature plasmas, a positive potential with respect to it. Therefore, the initially cold, i.e. gas temperature, ions can be accelerated and fall back onto the spacecraft, which often carry sensitive instruments, solar panels and other subsystems that might be disturbed or degraded by the unintended backflow from the applied thruster.

Hence, plasma diagnostics of the plasma surrounding a spacecraft that is created by the electric thruster and its surface modifying effects are necessary and have to be compared to ground-based experiments in laboratory. In particular, the diagnostics of electrons and ions as well as the fluxes of charged and neutral species toward plasma-facing surfaces by non-optical methods will be discussed. The focus is laid on ion beam diagnostics by Langmuir probes (LPs), Faraday cups (FCs) and retarding field analyzers (RFA), as well as by calorimetric and force probes (CPs, FPs) [2-4] in the test environment HIBEx. Also, on-board measurments at the satellite “Heinrich Hertz” which are performed by a designed diagnostic package (EPDP, see Fig.1) including a retarding potential analyzer, a plane Langmuir probe, and an erosion sensor will be presented [2].

Keywords: plasma diagnostics, probe measurements, electric spacecraft propulsion, ion thrusters, sputtering, charge-exchange collisions

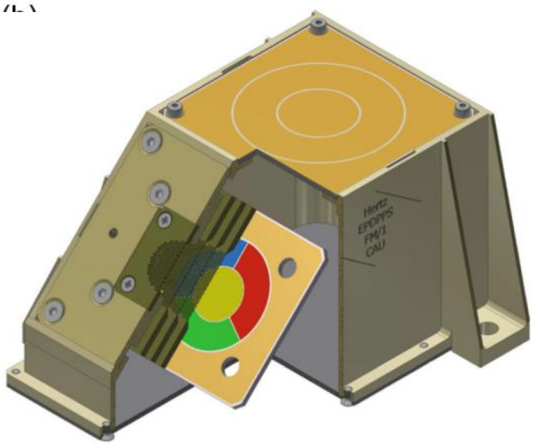


Figure 1: Plasma sensor of the EPDP [2]. The view into the sensor shows the four grids and the segmented collector of the RFA and the planar LP on top.

References:

1. Caruth, M.R., Gabriel,S.R., Kitamura, S., “Ion thruster charge-exchange plasma flow”, AIAA paper 82-0403, 1982.
2. Trottenberg, T., Bansemer, F., Böttcher, S., Feili, D., Henkel, H., Hesse, M., Kersten, H., Krüger, T., Laube, J., Lazurenko, A., Sailer, D., Schuster, B., Seimetz, L., Spethmann, A., Weis, S., Wimmer-Schweingruber, R., „An in-flight plasma diagnostic package for spececraft with electric propulsion“, Eur. Phys. J. TI Vol. 8, 16, 2021.
3. J. Benedikt, H. Kersten, A. Piel, “Foundations of measurement of electrons, ions and species fluxes toward surfaces in low-temperature plasmas”, Plasma Sources Sci. Technol., Vol.30, 033001, 2021.
4. Spethmann, A., Trottenberg, T., Kersten, H., Hey, F.G., Grimaud, L., Mazouffre,S., Bock, D., Tajmar, M., „Force probes for development and testing of different electric propulsion systems”, Eur. Phys. J. TI Vol. 9, 4, 2022.

Introduction of Anodic Oxide Film Formation Technology of Light Metals

Sungmo Moon [1,2,*]
[1] Nano-Surface Materials Division, Korea Institute of Materials Science, Changwon, South Korea
[2] Advanced Materials Engineering, Korea University of Science and Technology, Daejeon, South Korea

Abstract:

Formation of anodic oxide films on light metals, such as Al, Mg and Ti has been one of typical surface finishing methods which can be used for improving the resistances to corrosion and abrasion, surface appearance, bioactivity and adhesion with paints etc.. There are three different types of anodic oxide films; barrier type, porous type with regular cylindrical pores aligned vertically and porous type with irregular size and shape of pores, as shown in Fig. 1. Two types of anodic films of barrier type without pores and porous type with regular cylindrical pores are formed by migraton of ions through the oxide film under applied high electric field which is called anodizing method. Porous type of anodix oxide films with irregular size and shape of pores can be formed by dielectric breakdown of the anodic oxide films together with generation of micro-arcs, which is called plasma electrolytic oxideation method. The anodic oxidation can form thick oxide films on light metals by electrochemical reactions under the application of electric current and voltage between the working electrode and counter electrode. The anodic oxide film thickness is limited by how fast ions can migrate through the anodic oxide film for anodizing films or by how uniformly the applied electric field can cause dielectric breakdown over the surface for PEO films. Growth behavior of anodic oxide films on light metlas is dependent on various factors such as alloy composition, electrolyte composition, forms of applied current and electrolyte temperature. Current efficiency during anodic oxide film growth is determined by oxygen evlution and metal dissolution reactions. In this presentation, anodic oxide growth mechanism on varuous light metals of Al, Mg and Ti alloys will be discussed in view of ionic migration through anodic film, dissolution or breakdown of anodic film, arc generation and anodic film formation/growth under the application of high anodic electric field in various electrolytes.

Keywords: Anodic oxide film, Anodizing, Plasma electrolytiv oxidation, nanotube, Aluminum alloy, Magnesium, Titanium.

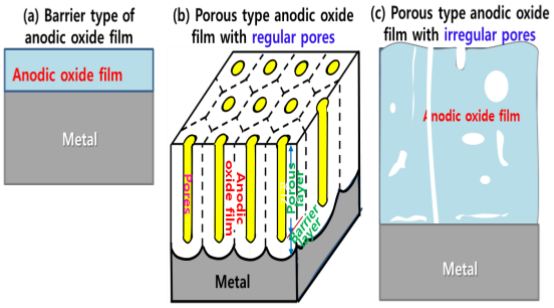


Figure 1: Schematic representation of three different types of anodic oxide films : (a), barrier type anodic oxide film; (b), porous type anodic oxide film; (c), PEO (plasma electrolytic oxidation) film.

Reference:

1. Moon S. (2018), “Anodic oxidation treatment methods of metals”, *J. Kor. Inst. Surf. Eng.*, 51, 1-10.

SICT 2024 Session I. A:
Surface treatments and coatings deposition and functionalization / Characterization / Properties Multifunctional composite and hybrid coatings

Structural Modification of Electrodeposited Cu 3D Structures for the Enhancement of CO_2 Cathodic Reduction

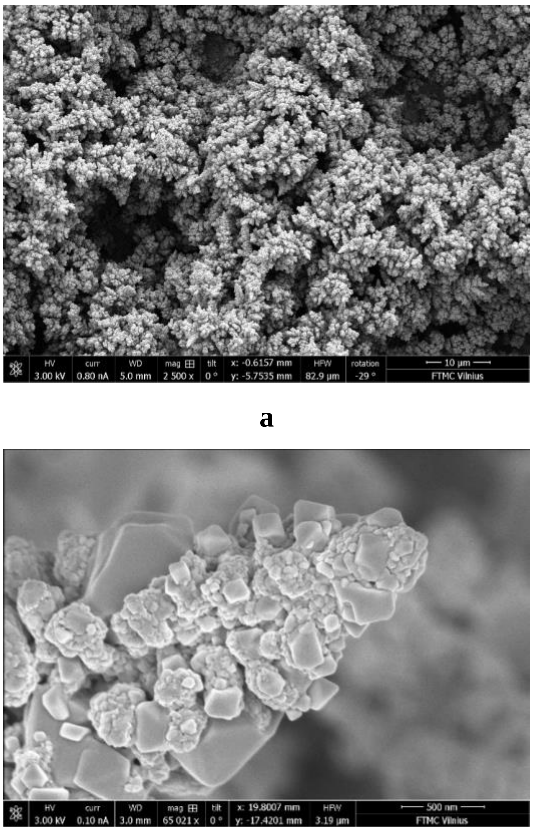
B. Serapiniene [1], A. Selskis[1], L. Gudaviciute[1], J. Juodkazyte[1], R. Ramanauskas [1*],
[1] Department Chemical Engineering and Technology, Center for Physical Sciences and Technology
Vilnius, Lithuania

Abstract:

An exclusive Cu surface feature has the ability to convert CO_2 into hydrocarbons with significant faradaic efficiency [1]. Simple polycrystalline Cu electrodes have a rather small surface area and therefore their efficiency is low. Three-dimensional nano-ramified Cu electrodes or foams can be produced by metal electrodeposition accompanied by intense hydrogen evolution. Such structures have a high electrochemically active surface area and are widely used in many applications, including CO_2 reduction. The catalytic activity of the metal is highly sensitive to surface morphology and structure, including lattice planes. Various strategies have been used to modify Cu to improve the current density and selectivity of CO_2 reduction to C2+ products [2]. It is known that Cu (1 0 0) and Cu (1 1 0) domains give superior selectivity for C2+ products, while Cu (1 1 1) promotes methane production [3]. Considering the importance of Cu microstructure for CO_2 reduction, we turned our attention to modify the distribution and facet ratio of Cu 3D structures.

The SEM image of the Cu foam (Fig. 1a) shows that it is composed of branched dendrites extending in all directions, while the detailed microscopic features (Fig. 1b) indicate that the Cu crystallites in a wall are grape-like deposits. Furthermore, these 3D Cu structures did not show the dominant crystallographic orientation. We tailored the distribution and facet ratio of the Cu 3D structures by electrochemical oxidation and reduction of the surface in chloride solution. The results show that the development of textured Cu electrode is accompanied by changes in the topography, which was followed by underpotential deposition (UPD) of Pb. The results obtained suggest that this treatment reduces the number of (111) sites and induces the growth of (100) and (110) domains. It has been shown that voltametric Pb UPD is a valuable tool for the characterisation and determination of single facet domains in multifaceted Cu surfaces.

Keywords: Cu 3D structure, CO_2 electroreduction, underpotential deposition, electrochemical faceting



a

b

Figure 1: SEM images of electrodeposited Cu 3D structures.

References:

1. Xie, H., Wang, T., Liang, J., Li, Q., Sun, S. (2018), Cu-Based Nanocatalysts for Electrochemical Reduction of CO_2, *Nano Today*, 21, 41–54.
2. Durand W. J., Peterson A.,Studt F.,Abild-Pedersen F., Norskov J. (2011), Structure effects on energetics of the electrochemical reduction of CO_2 by copper surfaces, *Surf. Sci.*, 605, 1354-1359.
3. Qiao Y., Seger B. (2024), *Current Opinion in Chem. Engin.*, 43, 100999.

Novel Industrial Sputtering Technology with Enhanced Flux Fraction of Deposited Material

J. Klusoň[1], M. Učík[1], M. Jílek[1], J. Hnilica[2], P. Klein[2], P. Vašina[2], H. Bolvardi[3], A. Lümkemann[3]
[1] PLATIT a.s., Šumperk, Czech Republic
[2]Department of Plasma Physics and Technology, Masaryk University, Brno, Czech Republic
[3]PLATIT AG, Selzach, Switzerland

Abstract:

PLATIT presents a novel sputtering technology F-Type which enables to achieve significantly enhanced power densities on the target surface and therefore significantly enhanced ionized flux fraction of deposited material too. From this point of view the technology can be taken as a possible industrial alternative to HiPIMS.

The new technology F-type will be compared with PLATIT conventional DC sputtering technology SCIL®. Common base for SCIL and F-Type is the central rotating cylindrical cathode with very efficient target cooling. Whereas in the case of SCIL the magnetic field design corresponds to more or less standard cylindrical arrangement, F-Type is equipped with a specific moveable magnetic core. As the core is periodically moving along the cathode axis, the total applied power is instantaneously concentrated into the limited area of the target in the vicinity of this core. It results in the possibility to reach very high power densities, especially for industrial DC magnetron sputtering. For the delivered power of 25 kW the maximal value of power density goes up to 800 W/cm^2, what is comparable with typical HiPIMS conditions.

F-Type technology was successfully used for the synthesis of hard protective AlCrN coatings. Examples of promising field test results will also be shown.

Keywords: PVD coatings, magnetron sputtering, power density, ionized flux fraction

Chemical vapour deposited SiOxNyCz thin Films for Advanced Barrier Solutions: Probing Chemical and Mechanical properties for better Performance

Farah Inoubli* [1], Babacar Diallo[1], Konstantina Cristina Topka[4], Thierry Sauvage[1], Raphael Laloo[3], Brigitte Caussat[2], Viviane Turq[3], Nadia Pellerin[1]

[1] CEMHTI-CNRS, Université d'Orléans France
[2] LGC Toulouse France
[3] CIRIMAT UPS Toulouse France
[4] Air Liquide Laboratories, Innovation Campus Tokyo Yokosuka City, Japan

Abstract

Despite of its high chemical inertia, Glass still interacts when exposed to aqueous solution. This reactivity could be problematic when it concerns particularly food and medicines containers. Thus, one of the biggest challenges that pharmaceutics and food industries are facing consist of limiting this interaction. But how?

In this work, we expose very promoting results on chemical vapour deposited silicon oxycarbonitride coatings in terms of chemical resistance in front of extremely aggressive aqueous solutions.

Different precursors were used leading to various film compositions with tunable properties. Pure silica films were obtained from tetraethylorthosilicate (TEOS) precursor. However, tris(dimethylsilyl)amine (TDMSA) and a novel proprietary trisilylamine-derivative precursor (TSAR) developed and provided by Air Liquide led to silicon oxycarbonitride films with different oxygen, nitrogen and carbon contents, depending on the deposition parameters (precursor, gas flow rates ratios and deposition temperatures td).

Films deposited on the two sides of a flat silicon monocrystalline substrate were subjected to moderately long alteration of one month in a citric acid aqueous solution with pH adjusted to 8 and under thermal conditions of 80°C. Their chemical resistance was assessed by tracking the structural evolution, the changes in the elemental composition and the calculation of the dissolved thickness if it exists. A wide range of characterization techniques were used for this purpose, namely ion beam analysis such as ERDA, RBS and NRA techniques, FTIR spectroscopy, XPS. SEM and AFM imaging techniques were also used to explore the changes occurring to surface state of our layers after exposure to the aqueous solution. Finally, nanoindentation tests have been done to verify any alteration happening to the hardness and the elasticity of the films. Very promising results were found especially for films both concentrated in N and C with a very high corrosion resistance even in such extreme chemical and thermal conditions.

To be closer to reality, pharmaceutical type I glass vials that have been successfully coated with SiOxNyCz thin layer, were tested according to the severe screening conditions of the United States Pharmacopeia USP<1660> chapter. They withstood to the test by preventing the degradation of the glass matrix with an average improvement factor of about 95% compared to a bare vial.

This excellent performance can make these materials a real key for the future of the pharmaceutical industry and can be transferable to multiple applications of surface coating by adaptation of the deposition conditions.

Keywords Silicon oxycarbonitride thin film, CVD, IBA, FTIR spectroscopy, chemical durability, nanoindentation, aqueous alteration mechanism.

References

1. Topka, K. C. et al. Critical level of nitrogen incorporation in silicon oxynitride films: Transition of structure and properties, towards enhanced anticorrosion performance. ACS Appl. Electron. Mater. **4**, 1741−1755 (2022).
2. Diallo, B. et al. Network hydration, ordering and composition interplay of chemical vapor deposited amorphous silica films from tetraethyl orthosilicate. J. Mater. Res. Technol. **13**, 534-547 (2021).
3. Topka, K.C. et al. Silicon oxynitride coatings are very promising for inert and durable pharmaceutical glass vials. ACS Applied Engineering Materials. https://doi.org/10.1021/acsaenm.3c00584

The impact of growth conditions on the crystalline quality and surface morphology of epitaxial III-N thin films: A molecular dynamics study

A. Zaiter, V. Hounkpati, G. Clavier, P. Ruterana, J. Chen
Centre de Recherche sur les Ions, les Matériaux et la Photonique UMR 6252, CNRS-ENSICAEN-UCBN-CEA, 6 Boulevard du Maréchal Juin, 14050 Caen Cedex, France

Abstract:

Semiconductor III-N nitrides such as indium nitride (InN), gallium nitride (GaN), and aluminum nitride (AlN) are widely used in optoelectronic and electronic devices such as light emitting diodes (LEDs), laser diodes (LDs), ultraviolet (UV) detectors, due to their thermal and chemical stability allowing them to withstand challenging operating conditions, including high temperatures, high voltages and currents.

The high quality III-N thin films are of great significance for increasing the lifetime and performance of III-N device. By using the molecular dynamics (MD) with an empirical Stillinger-Weber potential, we studied the impact of various growth conditions on the crystalline quality of III-N films at the atomic scale, such as temperature, growth rate and incident energies. The analysis focused on the crystal structure, surface morphology and internal stress of deposited films under these growth conditions.

Chitosan Functionalized Hexagonal Boron Nitride Nano sheets as Reinforcements for the Improvement of Anticorrosive Epoxy Coatings on AA2024 Al Alloy in Saline Medium

A.Madhan Kumar
Interdisciplinary Research Center for Advanced Materials, King Fahd University of Petroleum & Minerals, Dhahran, Kingdom of Saudi Arabia

Abstract:

Aluminum (Al) and its alloys have been largely employed as the most structural material in several components of the automobiles, marine structures, aerospace and defense and domestic appliances. The noble mechanical characteristics, along with the low weight, are the most vital benefits of this kind of metallic materials. However, limited corrosion resistance causes huge waste of resources, environmental impairment and economic losses that further restrict their applications. One of the most efficient strategies for protecting metallic materials against corrosion is to apply anticorrosive polymeric coatings. Epoxy coatings have been utilized as protective coatings for metallic structures exposed in atmospheric, marine and industrial sectors for the last 5 decades [1]. It can be utilized as a single primer layer and as a base coat in multi coat systems. Though epoxy coatings have been subjected to vast improvement for the past few decades, still there are some shortcomings that are not completed solved such as the less UV resistance, inadequate abrasion resistance and insufficient multi-functional characteristics including super hydrophobicity, antifouling, self-healing etc. These issues can be the productive approach in concentrating the development on epoxy coatings to get the desired features.

Two-dimensional hexagonal boron nitride (h-BN) nanosheets have gained abundant consideration due to their distinctive characteristics and widespread applications. However, the absence of adequate reactive functional sites on the surface together with the very stable structure limits their further application [2,3]. Thus, in the present study, we functionalized the HBN nanosheets with the chitosan through the simple chemical route and then utilized the functionalized HBN as viable reinforcement for epoxy coatings to improve their anticorrosion performance on AA2024 Al alloy in NaCl medium. The BN surface will be functionalized with hydrazine. Systematic structural, surface characterization and wettability will be performed to analyze the prepared epoxy nanocomposite coatings. Adhesion strength and corrosion protection performance of the prepared coatings will be also investigated in 3.5% NaCl solution through salt spray tests as well electrochemical corrosion measurements.The successful execution of this possible plan will enable us to develop these materials locally as the only step left would be to apply them for the protection of Al structures in real exposure condition (of course, after passing all required testing to be used in real exposures to the marine environment).

Keywords: Epoxy coatings; corrosion mitigation; AA2024 Al alloy.

References:

1. A. Madhan Kumar, Jobin Jose, Mohamed A. Hussein, Novel polyaniline/chitosan/reduced graphene oxide ternary nanocomposites: Feasible reinforcement in epoxy coatings on mild steel for corrosion protection,Progress in Organic Coatings, Volume 163, 2022, 106678.
2. Mingjun Cui, Siming Ren, Songlv Qin, Qunji Xue, Haichao Zhao, Liping Wang, Processable poly(2-butylaniline)/hexagonal boron nitride nanohybrids for synergetic anticorrosive reinforcement of epoxy coating, Corrosion Science, Volume 131, 2018, Pages 187-198.
3. Fushen Lu, Chenqi Liu, Zihua Chen, Udayakumar Veerabagu, Zhaobin Chen, Mingen Liu, Liangsheng Hu, Hong Xia, Limei Cha, Wenjing Zhang, Polypyrrole-functionalized boron nitride nanosheets for high-performance anti-corrosion composite coating, Surface and Coatings Technology, Volume 420, 2021, 127273.

Enthalpy-driven self-healing in thin metallic films on flexible substrates

C.O.W. Trost1, P. Kreiml1, T. Jörg2, V. Terziyska2, C. Mitterer2, M. J. Cordill1,2, A. Lassnig11
[1] Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Jahnstrasse 12, Leoben 8700, Austria
[2] Department of Materials Science, Montanuniversität Leoben, Jahnstrasse 12, Leoben 8700, Austria

Abstract:
Self-healing microelectronics are needed for costly applications with limited or no access for repairs. They are needed in fields such as space exploration to increase lifetime and decrease both costs and the environmental impact. While advanced self-healing mechanisms for polymers are numerous, practical ways for self-healing in metal films have yet to be found. A concept for an intrinsic autonomous self-healing metallic film system was developed, allowing the healing of cracks in metallic films on flexible substrates. The concept relies on stabilising metastable thin films with high mixing enthalpy via segregation barriers. The phase-separating tendency of the binary alloy [1] is exploited to heal cracks introduced by straining above the crack onset strain. This allows the films to possess intrinsic autonomous self-healing capabilities triggered by cracking at temperatures not detrimental to flexible microelectronics. The effect will be shown on metastable binary alloy thin films, stabilised via a segregation barrier. Without a segregation barrier, the system is known to exhibit spontaneous particle formation on the surface. This property was controlled and directed to heal cracks and partly restore the electro-mechanical properties of the multilayer system. This mechanism opens up the field of thin metallic self-healing films that could severely impact the design of future microelectronics.

Keywords: •self-healing, thin films, flexible substrates. Microelectronics, metastable alloy

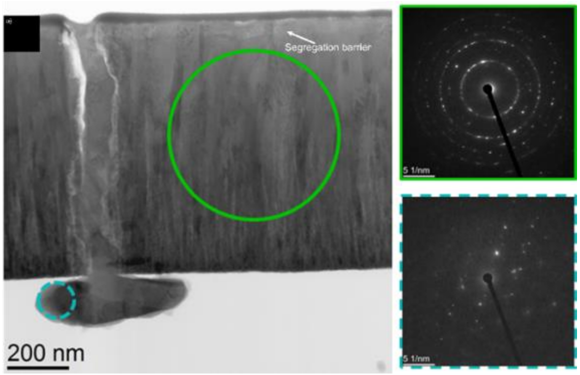


Figure 1: Transmission Electron image of a healed crack in a metastable binary alloy.

References:

1. W. S. Cunningham et al., “Unraveling Thermodynamic and Kinetic Contributions to the Stability of Doped Nanocrystalline Alloys using Nanometallic Multilayers,” Advanced Materials, vol. 34, no. 27, e2200354, 2022, doi: 10.1002/adma.202200354.

Comparison of nitriding behaviour for austenitic and duplex stainless steel with precipitation hardening steel

D. Manova [1], S. Mändl [1,*]
[1] Leibniz Institute of Surface Engineering (IOM), Leipzig, Germany

Abstract:

Nitriding of austenitic stainless steel is nowadays an established process. Here, the formation of expanded austenite with a lattice expansion of up to 12% and with an incorporated nitrogen content of up to 30 at.% is observed. Nevertheless, using in-situ X-ray diffraction (XRD) during the nitriding with low energy nitrogen ions allows us to collect information crucial to the understanding of the underlying physical processes. For example, a direct observation of the evolution of the total layer thickness with time (i.e. nitriding velocity) and the evaluation of the depth-integrated lattice expansion is possible. Furthermore, in-situ XRD during argon ion sputtering of the surface allows us to obtained depth resolved data with a resolution better than 50 nm.

Here, we compare the nitriding behavior as function of time (and depth) for an austenitic stainless steel 1.4571, a super-austenite 1.4539, a duplex steel 1.4462 and precipitation hardening steel 1.4545.

Both austenitic steels exhibit a similar evolution of the layer thickness with time and temperature while the super-austenite presents a much higher lattice expansion for the (200) oriented grains. The thermal stability of the expanded austenite is also quite similar, with the formation of CrN precipitates starting around 475 – 500 °C.

In the duplex steel, a much faster nitrogen diffusion is observed for the ferritic grain compared to the austenite, yet no formation of expanded ferrite is observed by XRD. As the increased nitrogen content is stabilizing the austenitic phase, a transition from ferrite towards austenite with increasing nitrogen incorporation is postulated. At the same time, the onset of CrN precipitation is shifted towards lower temperatures by about 20 K, presumably caused by a microstructure containing smaller grains and more defects or grain boundaries.

For the precipitation hardening steel, a very interesting behaviour was found. In addition to a very fast nitrogen diffusion with more than 5 µm reached in less than 10 minutes at 400 °C, a very peculiar evolution of the microstructure was observed. Even at 350 °C – much below the published transition temperature for precipitation hardening – a complete loss of diffraction intensity is observed for the whole measured range of 35° – 54° in 2θ after 30 minutes. For higher temperatures, a broad peak corresponding to a martensitic phase appears, indicating a rather small grain size. Yet no diffraction peaks corresponding to a nitrogen containing phase are observed. The very strong variation of the results with nitriding temperatures indicate that special care is necessary when using a commercial nitriding process to obtain consistent results.

Keywords: nitriding, stainless steel, in-situ XRD, expanded austenite.

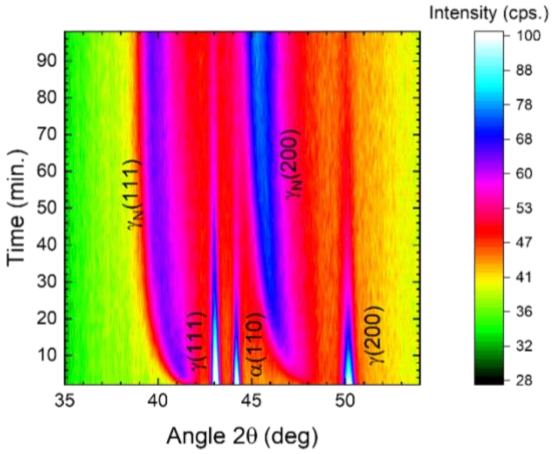


Figure 1: Contour plot of in-situ XRD data obtained during nitriding of the duplex steel 1.4462 at 400 °C.

References:

1. Manova, D., S. Mändl, S. (2019), Perspectives: In-situ XRD measurements to explore phase formation near surface region, *J. Appl. Phys.*, 126, 200901.
2. Manova, D., Schlenz, P., Mändl, S. (2022), A combination of ion beam sputtering and in situ x-ray diffraction as a method for depth-resolved phase analysis using nitrogen-implanted austenitic stainless steel as an example, *J. Appl. Phys.*, 131, 025306.

Challenges on Surface Oxidation of Steel – Alloying Elements and Scale Behavior

K. Anand [1,*], A. Koltsov [1], L. Turri [1], J. Scheid [1], P. Alexandre [1]
[1] ArcelorMittal R&D Maizieres, Maizieres les Metz, France

Abstract:
In steelmaking process metallic surfaces interact with oxidizing environment (air, water) at high temperatures, leading to the formation and growth of different types of oxides. Steel surface is one the most reactive metallic alloyed surfaces where mostly Fe atoms transfer electrons to the O atoms, but alloying elements and other oxidant groups such as hydroxyl (-OH) can complexify the mechanism, that requires a more in-depth thermodynamic and kinetic study. Another dimension of questions come up due to different process conditions varying temperature ranges, oxidant types and the exposure time. For example, high oxidation in reheating furnace is extremely different from the thin oxidation layers formed in continuous annealing step before hot-dip galvanizing. Oxidation kinetics and related thermodynamics depends on the process conditions as well as surface chemistry of the Steel surface. Here, we present oxidation phenomenon in reheating furnace focusing on the surface structural aspect of oxide layers formed and how their structure evolves with process conditions (Figure 1). Preliminary results show the oxide layer decomposition changes due to the cooling conditions. Also, interfacial structure is altered by the presence of alloying elements which in turn influences scale adhesion and its surface properties. Even the impact on de-scaling parameters can be envisaged. Avoiding details, we briefly highlight the open questions in this area and present a perspective of understanding the surface mechanism using both experimental and computational tools further to disentangle key parameters and factors involved in oxidation process.

Keywords: surface oxidation, Steel, re-heating furnaces, microstructural experiments, Industrial processes

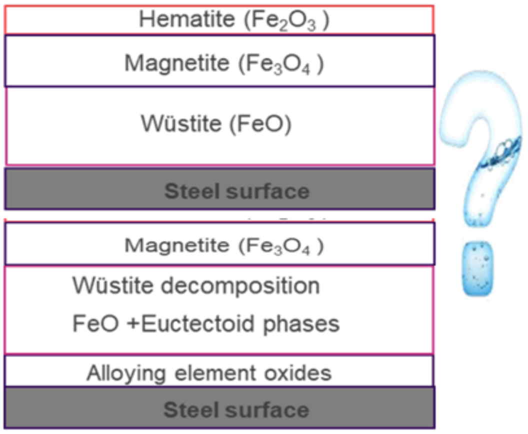


Figure 1: Schematic illustrating two different morphological arrangement of oxide layers and the related interfacial interplay which requires a more systematic understanding of surface chemistry of Steel and its interaction with the oxidizing environment.

References:

1. Takeda, M. et al. (2010), Influence of Oxidation Temperature and Cr Content on the Adhesion and Microstructure of Scale on Low Cr Steels. Oxid. Met., 73, 1–13.
2. Osei, R. et al. (2022). Effect of Cu Additions on Scale Structure and Descaling Efficiency of Low C Steel Reheated in a Combustion Gas Atmosphere. Oxid. Met., 98, 3–4

Influence of current density and process time on structural, chemical and physical behaviour of anodized aluminum

Lea Breu [1,2*], Martina Bubrin [1], Timo Sörgel [2]
[1] Robert Bosch Manufacturing Solutions GmbH, Stuttgart, Germany
[2] Center for Electrochemical Surface Technology ZEO, Aalen University of Applied Sciences, Aalen, Germany

Abstract:
Anodization of aluminum is a useful process to improve the corrosion resistance of aluminum. For industrial application the process time is an important parameter due to production costs.. However, the properties must not be altered. In this study pouros aluminia is produced on 6061 aluminum alloy with an oxalic acid electrolyte to investigate the impact of the current density and the process time to the microstructure and the corrosion resitance. To speed the process up, a wide range of current density is investigated. To determine corrosions resistance and microstructure of the layer, electrochemical impedance spectroscopy (EIS) and scanning electron microscopy (SEM) are used, respectively. We can see that with a high current density, process time is reduced, while similar structures can be achieved (figure 1). Pore diameter and pore length are similar. The microstructure can be described as corral-like. Contrary to other literature, there is no change in the porosity of the layer due to higher current density [1]. At higher current density, microstructure of the layer changes to longer and larger pores. The effect of this change on corrosion resistance will be investigated.
Further we will investigate hydrogen permeation through the anodizing layers. With the improvement of coating resistance, there is an impact on hydrogen permeation. To investigate the hydrogen permeation, an electrochemical Devanathan-Stachurski-cell is used.

Keywords: anodization, oxalic acid, high current density, corrosion resistance, hydrogen permeation, electrochemical impedance spectroscopy.

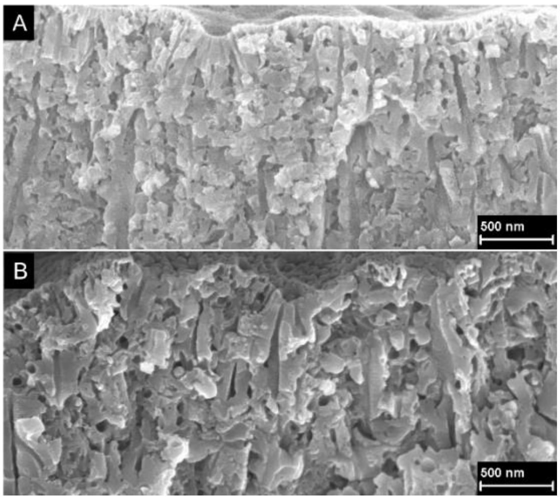


Figure 1: Figure shows a similar microstructure demonstrated via cross section SEM pictures of oxalic acid anodization layers produced with A): 1 A/dm² current density B): 5 A/dm² current density.

References:

1. Chung, I.C., Chung, C.K., Su, Y.K. (2017), Effect of current density and concentration on microstructure and corrosion behavior of 6061 Al alloy in sulfuric acid, *surf. coat. technol.*, 313, 299-306.
2. Cabral-Miramontes, J., Gaona-Tiburcio, C., Estupinán-López, F., Lara-Banda, M., Zambrano-Robledo, P., Nieves-Mendoza, D., Maldonado-Bandala, E., Chacón-Nava, J., Almeraya-Calderón, F. (2020), Corrosion Resistance of Hard Coat Anodized AA 6061 in Citric–Sulfuric Solutions, *Coatings* 10, 601

Adhesion improvements of paint on polypropylene copolymers by flame and UV-ozone surface treatments

M. Borg[1,2], Y. Tison[1], K. Lavisse[2], C. Derail[1]
[1]Universite de Pau et des Pays de l'Adour, E2S UPPA, CNRS, IPREM, Pau, France
[2] Renault Group, Ingénierie Process Injection Plastic et Peinture, Guyancourt, France

Abstract: Polymers based on polypropylene copolymers (PP) are widely used by automotive industry because of their suitable properties as ability to deform, low cost, high shock resistance, … However, PP are known for their low surface tension which leads to low adhesion of paint. Surface preparation like flame and UV-ozone treatments are necessary to improve paint adhesion.[1-4] A study by Landete-Ruiz *et al.*[3] has shown that UV-ozone treatment on ethylene-vinyl acetate (EVA) copolymer surfaces increases wettability, created oxygen fragments and modifies roughness. They demonstrated that the vinyl acetate content, which has an effect on the crystallinity of the copolymer, induces an impact on efficiency of surface treatment to improve adhesion. In this work we propose a complete characterization, at different scales, of a series of compounds based on PP with a variable ratio of a mineral filler to propose adhesion's mechanisms of paint on plastics. Flame and UV-ozone model treatment were specifically chosen to activate surface. Surface characterizations such as XPS, SEM, AFM and contact angle measurements are used to understanding interface mechanisms. We demonstrate the poor effect of the filler ratio onto various parameters linked to the adhesion properties although physico-chemical parameters are affected. In Figure 1 we present the evolution of crystallization degree's and in parallel the surface tension values as a function of filler's ratio. Specialized testing (high-pressure cleaning resistance, reference grid cutting adhesion test), as presented in Figure 2, are used to evaluate adhesion according to the filler ratio into PP. To conclude, we demonstrate that crystallinity degrees and filler ratio have no impact on adhesion properties. Other characterizations in progress should complete these observations.

Keywords: adhesion, surface treatment, polypropylene, flame treatment, UV-ozone treatment, surface characterizations, paint.

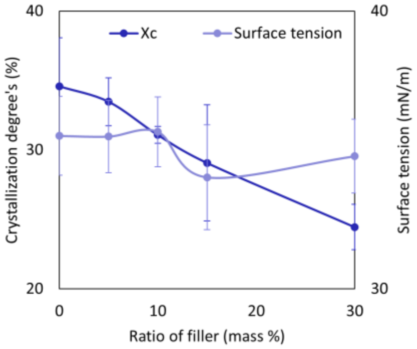


Figure 1: evolution of crystallinity and surface tension as a function of filler ratio.

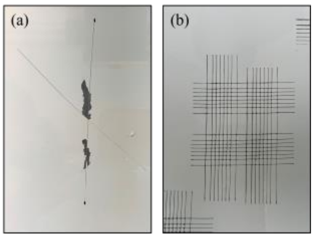


Figure 2: illustration of high-pressure cleaning resistance (a) and grid cutting adhesion test (b) onto paint surfaces of the study.

References:

1. Farris, S., Pozzoli, S., Biagioni, P., Duo, L., Mancinelli, S., & Piergiovanni, L. (2010). The fundamentals of flame treatment for the surface activation of polyolefin polymers - A review. *Polymer, 51*, 3591-3605.
2. Gongjiang, B., Yunxuan, W., & Xingzhou, H. (1996). Surface modification of polyolefine by UV light/ozone treatment. *Journal of Applied Polymer Science, 60*, 2397-2402.
3. Landete-Ruiz, M., & Martin-Martinez, J. (2015). Improvement of adhesion and paint ability of EVA copolymers with different vinyl acetate contents by treatments with UV-ozone. *Int. Journal of Adhesion and Adhesives, 58*, 34-43.
4. Sutherland, I., Brewis, D., Heath, R., & sheng, E. (1991). Modification of polypropylene surfaces by flame treatment. *Surface and interface analysis, 17*, 507-510.

Transparent PDMS Surfaces with Covalently Attached Lubricants for Enhanced Anti-Adhesion Performance

Tanja Eder,[a,b] Andreas Mautner,[b,c] Yufeng Xu,[a] Michael Reithofer,*[d] Alexander Bismarck,*[b,e] Jia Min Chin*[a]

[a] Department of Functional Materials and Catalysis, University of Vienna, Währinger Straße 42, 1090 Vienna, Austria
b Institute of Materials Chemistry and Research, University of Vienna, Währinger Straße 42, 1090 Vienna, Austria
c Institute of Environmental Biotechnology, University of Natural Resources and Life Sciences (BOKU), Konrad-Lorenz-Straße 20, 3430 Tulln a.d. Donau, Austria
d Institute of Inorganic Chemistry, University of Vienna, Währinger Straße 42, 1090 Vienna, Austria
e Department of Chemical Engineering, Imperial College London, South Kensington Campus, London, SW7 2AZ, UK.

Abstract:
Liquid-like surfaces featuring slippery omniphobic covalently attached liquids (SOCALs) reduce unwanted adhesion by providing a molecularly smooth and slippery surface arising from the high mobility of the liquid chains.[1] Such SOCALs are commonly prepared on hard substrates such as glass, wafers or metal oxides, despite the importance of non-polar elastomeric substrates such as polydimethylsiloxane (PDMS) in anti-fouling or non-stick applications. Compared to polar elastomers, hydrophobic PDMS elastomer activation and covalent functionalization is significantly more challenging, as PDMS tends to display fast hydrophobic recovery upon activation as well as superficial cracking.[2,3] Through extraction of excess PDMS oligomer and fine-tuning of plasma activation parameters, homogeneously functionalized PDMS with fluorinated polysiloxane brushes could be obtained whilst at the same time reducing crack formation. Polymer brush mobility was increased through the addition of a smaller molecular silane linker to exhibit enhanced de-wetting properties and reduced substrate swelling compared to functionalizations featuring hydrocarbon functionalities. Linear polymer brushes were verified by thermogravimetric analysis (TGA). The optical properties of PDMS remained unaffected by the activation in high-frequency plasma but were impacted by low-frequency plasma. Drastic decreases for solid adhesion of not just complex contaminants but even ice could be shown in horizontal push tests, demonstrating the potential of SOCAL-functionalized PDMS surfaces for improved non-stick applications.

Keywords: Non-stick coatings; Anti-icing; Polymer interfaces; Liquid repellence; Polymer brushes; Transparent surfaces

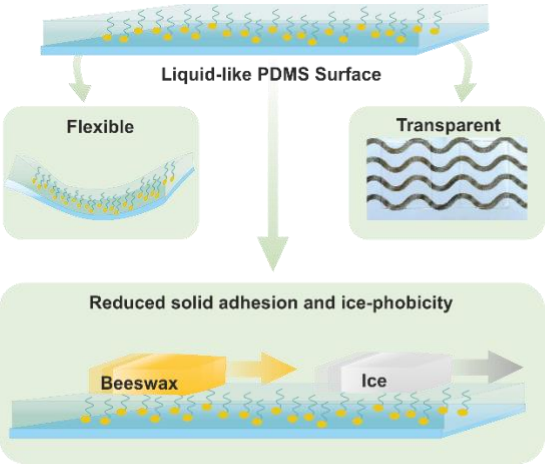


Figure 1: Covalent grafting of lubricants creates a liquid-like surface on PDMS and allows to impart anti-adhesion properties onto silicone substrates preserving their flexibility and transparency.

References:

1. Chen, L.; Huang, S.; Ras, R. H. A.; Tian, X. Nat Rev Chem 2023, 7, 123-137.
2. Befahy, S.; Lipnik, P.; Pardoen, T.; Nascimento, C.; Patris, B.; Bertrand, P.; Yunus, S. Langmuir 2010, 26, 3372-3375.
3. Wang, Z.; Luo, Y.; Zheng, F.; Zhang, N.; Yin, C.; Li, J.; He, C.; Peng, X.; Huang, Z.; Fang, P. Surface and Interface Analysis 2018, 50, 819-826.

Investigation of Thermal and Mechanical Properties of Ti-Si-B-C Nanocomposite Coatings

A. Thewes [1*], P.M. Reinders [1,2], H. Paschke [2], T. Brückner [2], C. Sternemann [3], M. Paulus [3], W. Tillmann [4], J. Urbanczyk [4], N.F. Lopas Dias [4]
[1] Institute for Surface Technology, TU Braunschweig, Braunschweig, Germany
[2] Fraunhofer Institute for Surface Engineering and Thin Films IST, Braunschweig, Germany
[3] Fakultät Physik/DELTA, Technische Universität Dortmund, Dortmund, Germany
[4] Institute of Materials Engineering, TU Dortmund, Dortmund, Germany

Abstract:

Nanocomposite Ti-Si-B-C coatings were analyzed regarding their hardness, fracture toughness, and oxidation resistance. These coatings were deposited by plasma enhanced chemical vapor deposition (PECVD). Self-organisation during coating deposition leads to a nanocomposite coating structure, consisting of nanocrystalline TiC grains embedded in an amorphous matrix. This coating system was deposited on nitride AISI H11 hot working steel and with a TiN interlayer and offered good adhesion (HF2). With a hardness of up to 29.0 GPa, the coating system is a possible candidate for application as a wear reducing coating on a tool surface. Given the harsh environment of many tribological applications in hot forming or molding, the thermal stability and oxidation resistance are of special interest. In-situ X-ray diffraction analysis at elevated temperatures showed the beginning of TiO_2 formation at 800 °C, marking it as the starting point for oxidation. With an increase in temperature, the TiO_2 reflexes get stronger, but (200) TiC is always the strongest reflex.

Keywords: Ti-Si-B-C, nanocomposite coating, PECVD, oxidation resistance

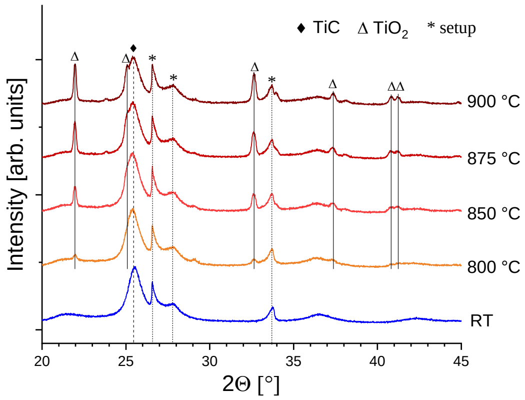


Figure 1: X-ray diffraction patterns of a Ti-Si-B-C coating at different temperatures are shown. TiO_2 reflexes appear at 800 °C, but are weakly pronounced. At 900 °C, TiO_2 reflexes are strong, but (200) TiC is still the strongest reflex. This is due to a thin oxide film on top of an otherwise intact Ti-Si-B-C coating.

References:

1. Mishra, S.K., Mahato, P., Mahato, B. Pathak, L.C. (2013) Thermal stability and effect of substrate temperature of TiSiBC hard nanocomposite coatings on microstructure, mechanical, thermal behavior deposited by magnetron sputtering, *Applied Surface Science*, 266, 209-213.
2. Mahato, P., Singh R.J., Mishra, S.K. (2016) Nanocomposite Ti-Si-B-C hard coatings deposited by magnetron sputtering: Oxidation and mechanical behavior with temperature and duration of oxidation, *Surface & Coatings Technology*, 288, 230-240.
3. Mahato, P., Banerjee, P., Murmu, M., Hirani, H., murmur, N.C., Mishra, S.K. (2021) Investigation on Multifunctional Properties of Sputtered Ti-Si-B-C Coating with Varied Thickness over Targeted Surface, *Journal of Materials Engineering and Performance*, 30, 4432-4444.

Core/shell Ge/Al quantum dot lattices in amorphous SiC: structure and photo-conversion properties

M. Tkalčević [1], I. Periša[1] M. Bubaš[1], S. Bernstorff [2]
G. Provatas[1], M. Mičetić[1]
[1] Ruđer Bošković Institute, Bijenička cesta 54, 10000 Zagreb, Croatia
[2] Elettra Sincrotrone S.C.p.A., Strada Statale 14, km 163.5 in AREA Science Park,
34149 Basovizza/Trieste, Italy

Abstract:

Thin films containing self-assembled quantum dots (QDs) offer customizable properties through adjustments in QD size and spacing. These films were fabricated using magnetron sputtering deposition, a process where a target material is bombarded with high-energy ions in a vacuum chamber, causing atoms to be ejected and deposited onto a substrate to form a thin film.

Incorporating a metal shell around the QDs allows for precise control, essentially acting as a core. This configuration significantly influences the structural, optical, and optoelectronic characteristics of the system, introducing strain and stress on the QDs and affecting their size, shape, and crystalline structure. The size of the germanium (Ge) core and the thickness of the surrounding metal shell play crucial roles in determining the structural, electrical, and optical properties of Ge quantum dots (QDs). The metal shell serves various purposes, such as protection against oxidation and potential enhancement of optical properties through localized surface plasmon resonance. Adjusting the metal shell thickness can significantly alter thin film properties to suit specific applications.

To analyze the structural composition, we employed a combination of two ion beam techniques: Time-of-Flight Elastic Recoil Detection Analysis (TOF-ERDA) and Rutherford Backscattering Spectrometry (RBS). Structural characterization primarily relied on Grazing Incidence Small-Angle X-ray Scattering (GISAXS).

For optical characterization, spectroscopic ellipsometry was used and for electrical characterization including quantum efficiency (QE) measurements Photovoltaic Testing System from Sciencetech was used.

Keywords: Ge QDs, core-shell structure, GISAXS, magnetron sputtering, ion beam analysis, RBS, quantum efficiency

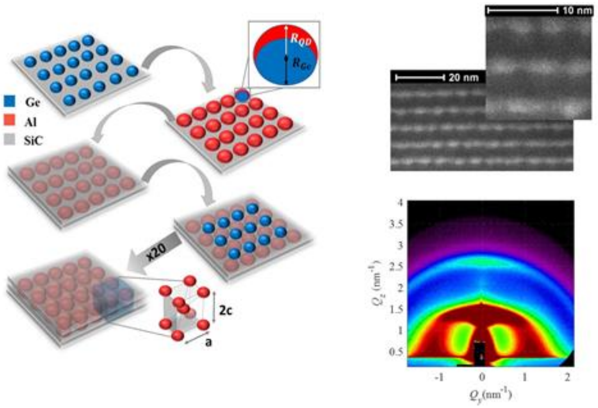


Figure 1: Proces of formation of germanium quantum dots (QDs) arranged in a body-centered tetragonal (BCT) lattice structure within a silicon carbide (SiC) matrix. The self-assembly of these QDs is confirmed through high-resolution transmission electron microscopy (HRTEM) and grazing incidence small-angle X-ray scattering (GISAXS)

References:

1. Periša, I., Tkalčević, M., Isaković, S., Basioli, L., Ivanda. M., Bernstorff, S., Mičetić, M. (2022) Ge/Al and Ge/Si_3N_4/Al core/shell quantum dot lattices in alumina: boosting the spectral response by tensile strain, *Materials*
2. Tkalčević, M., Basioli, L., Salamon, K., Šarić, I., Parramon, J. P., Bubaš, M., Bogdanović-Radović, I., Bernstorff, S., Fogarassy, Z., Balázsi, K., Petravić, M., Mičetić, M. (2020) Ge quantum dot lattices in alumina prepared by nitrogen assisted deposition: Structure and photoelectric conversion efficiency, *Sol. Energy Mater. Sol. Cells.*

Plasma Tech Session I. B: Plasma fundamentals / Modelling / Atomic and Molecular Processes

Plasma devices for satellite propulsion

N. Jochen Schein

Bundeswehr University Munich, Germany

Abstract:

Most space propulsion system are based on Newton’s third law, meaning that mass is accelerated to provide impulse in the opposite direction. Electric propulsion (EP) working with that principle is based on the creation of plasmas which are either fully or partially accelerated by -except for electrothermal systems- the application of electric or magnetic fields. The thus created high exit velocities allow for a reduction of propellant mass compared to chemical propulsion systems but also cause low thrust-to-power efficiencies. Hence the use of EP systems is limited and mission dependent, but it is also an enabling technology for certain aspects like the use of small satellites or for very fine pointing requirements. In this presentation -after some introductory remarks - three different propulsion systems are discussed, based on different ways of plasma production: the so-called vacuum arc thruster - which uses the cathode material as propellant-, the C-STAR -which is based on the magnetic acceleration of particles created in a capacitively coupled RF discharge, and the inductively coupled RF ion thruster - which has already been flown on many space missions.

The three systems will be introduced and the various issues with their different acceleration mechanisms will be discussed.

Langmuir probe measurements in a dual-frequency capacitively coupled rf discharge

V. Schneider[1], J. Schleitzer[1], Ihor Korolov[2], Gerrit Hübner[2], Peter Hartmann[3], Julian Schulze[2], and Holger Kersten[1]
[1] Institute of Experimental and Applied Physics, Christian-Albrechts-University Kiel, Germany
[2] Chair of Applied Electrodynamics and Plasma Technology, Faculty of Electrical Engineering and Information Sciences, Ruhr University, Bochum, Germany
[3] Institute for Solid State Physics and Optics, Wigner Research Centre for Physics, Hungary

Abstract:

A dual-frequency capacitively coupled rf argon plasma has been investigated using a passively compensated Langmuir probe. The discharge is driven by two different excitation frequencies (13.56 MHz and 27.12 MHz) simultaneously with variable phase angle θ between them, utilizing the electrical asymmetry effect (EAE) [1-3]. The EAE enables the control of the bias voltage and, thus, the ion energy almost independent on the ion flux by varying the relative phase between the two harmonics, while other discharge parameters are constant [1-3]. An extended characterization of this plasma is crucial for its optimization and improvement for application, both in research and industry, making its investigation with several diagnostic methods mandatory.

With a passively compensated Langmuir probe [4,5] the floating potential, plasma potential, electron temperature and electron density are measured for different phase angles ($0° - 180°$) in two different geometrically asymmetric discharges. Similar to the dc self-bias, the plasma parameters show a pronounced dependence on the phase (Fig. 1). Their general behaviour can be explained by phase-dependent sheath expansion dynamics as shown by PIC/MCC simulations, where beams of electrons are generated by the respective expanding sheath and accelerated into the plasma bulk, leading to phase-dependent electron temperature and density [1,6]. However, the measured profiles of the density and temperature as a function of phase in both experimental setups are not symmetric around $\theta = 90°$, unlike the dc self-bias. This observation is confirmed by PIC/MCC simulations, which reveal asymmetrical electron excitation/ionization dynamics at the corresponding phases [7]. This implies that the observed trends are a property of the 2f discharge in combination with a geometric asymmetry of the discharge.

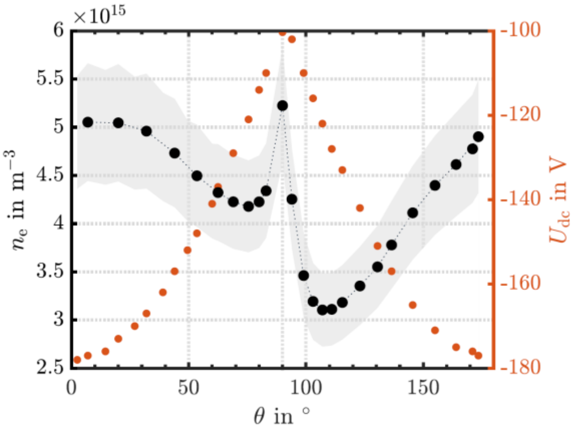


Figure 1: Electron density n_e together with the self-bias U_{dc} in dependence on the phase shift at a pressure of 3 Pa. The plasma parameters were measured 17 mm above the electrode. The error margins are indicated in grey.

Keywords: plasma diagnostics, radiofrequency discharges, plasma sheath, phase variation, Langmuir probe, electrical asymmetry effect, physical quantities

References:

1. B. G. Heil et al., Journal of Physics D: Applied Physics 41, 165202 (2008).
2. Z. Donkó et al., Journal of Physics D: Applied Physics 42, 025205 (2009).
3. J. Schulze et al., Journal of Physics D: Applied Physics 42, 092005 (2009).
4. A. Cantin and R. R. J. Gagné, Applied Physics Letters 30, 316–319 (1977).
5. A. Wendt, Review of Scientific Instruments 72, 2926–2930 (2001).
6. J. Schulze et al., Plasma Sources Science and Technology 19, 045028 (2010).
7. J. Schleitzer et al., Physics of Plasmas (2023), submitted.

Optically trapped microparticles in a dual-frequency capacitively coupled rf discharge

J. Schleitzer [1], V. Schneider [1] and H. Kersten [1]
[1] Institute of Experimental and Applied Physics, Christian-Albrechts-University, Kiel, Germany

Abstract:

Many different diagnostics can be used to measure the spatial distribution and temporal evolution of plasma parameters. Over the past decade, the concept of utilizing externally injected small microparticles as non-invasive probes, influenced by various forces and energy fluxes in plasmas, has been implemented. Information about local electric fields [1], energy fluxes toward the particles [2], and momentum transfer by ions to the particles [3] can be obtained based on their behavior in the surrounding plasma.

Especially the manipulation of microparticles by an optical tweezer is of great interest, as it enables the microprobe to be positioned in areas of the plasma that are typically inaccessible by conventional diagnostic methods, such as 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 almost separately based on the utilization of electrical asymmetries in the plasma [4,5]. It is generated by a superposition of two consecutive harmonics with variable phase angle between them.

The crucial parameter to measure when employing optical tweezers is the external force acting on the microprobe. This force is determined by observing the displacement of the particle within the optical trap. The confined microprobe is moved through the plasma and the sheath, thereby moving relative 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 gas pressure and phase angle between the two harmonics, the extent of the sheath, as well as the particle charge evolution within the sheath are determined [7].

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

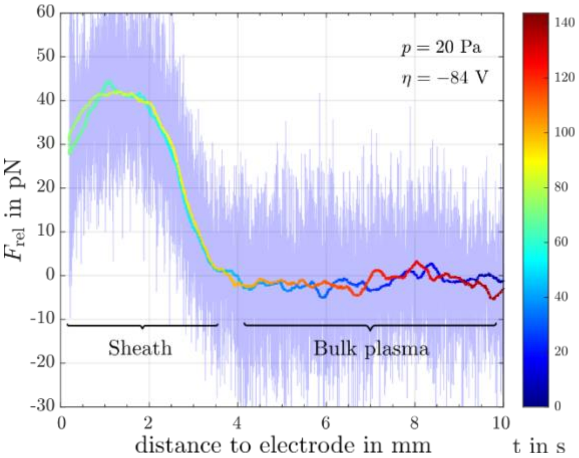


Figure 1: The graph shows the measured relative force profile obtained by the use of an optically trapped microparticle for a certain pressure p and a specific dc self-bias η in a dual-frequency discharge. The colored curve represents an averaging over the raw data shown and the color gradient illustrates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

A novel test-facility based on X-ray imaging and mm-wave polarimetry for plasma diagnostics

E. Naselli [1,*], G. Finocchiaro [1,2], G.S. Mauro [1], B. Mishra [1,2], B. Peri [1,3], A. Pidatella [1], G. Torrisi [1] and D. Mascali [1]
[1] National Institute for Nuclear Physics-Laboratori Nazionali del Sud (INFN-LNS), Catania, Italy
[2] Department of Physics and Astronomy, University of Catania, Catania, Italy
[3] Sicilian Center of Nuclear Physics and Physics of Matter (CSFNSM), Catania, Italy

Abstract:

At INFN-LNS, a non-invasive plasma multidiagnostics system has been developed in the frame of the PANDORA [1] project and synergically with the SAMOTHRACE ecosystem funded by the EU Next Gen Program, aimed at fundamental nuclear and plasma physics research. Magnetically confined plasmas generated in a powerful and unconventional superconducting ion source equipped with tens of detection and diagnostic devices (RF polarimeter, optical emission spectroscopy, X-ray imaging and space and time-resolved spectroscopy, RF probes, scope, HPGe detector array) can be properly investigated. High precision measurements of the thermodynamic plasma parameters (electron temperature and density) and of the plasma structure and elemental composition can be carried out.

Recently, within the synergic SAMOTHRACE (Sicilian MicronanoTech Research And Innovation Center) project frame, two new diagnostics testbenches – the PYN-HO prototype and the VESPRI 2.0 setup – were designed and they aims to develop and improve detectors and techniques beyond the state of art. The PYN-HO (Probing x-raYs by imagiNg and pin-Hole spectrOscopy) prototype is conceived to operate in four different configurations. It will allow to perform X-ray imaging and space-resolved spectroscopy (Fig.1, top) by CCD pin-hole camera setup [2, 3] and advanced algorithms for Single Photon-Counted (SPhC) and High-Dynamic-Range (HDR) analysis [4], with the related calibrations and characterizations, both on test-benches and in magnetically confined plasmas. Two PYN-HO configurations will be instead dedicated on high energy resolution diffractometric spectroscopy measurement in X-ray and optical domains basend on the use of gratings. The design of the VESPRI 2.0 mm-wave polarimeter [5] (Fig.1, bottom) is based on a heterodyne approach to measure the magnetoplasma-induced Faraday rotation, providing measurement of the plasma line-integrated electron density. The analysis method is based on the detection of Lissajous figure from a two channels scope in an x-y representation of a direct probing RF signals crossing the magnetoplasma.

Keywords: plasma non-invasive diagnostics, magnetically-confined plasmas, single-photon-counted X-ray imaging, space-resolved X-ray spectroscopy, pin-hole technique, mm-wave polarimeter, Faraday rotation evaluation, heterodyne approach, Lissajous figure

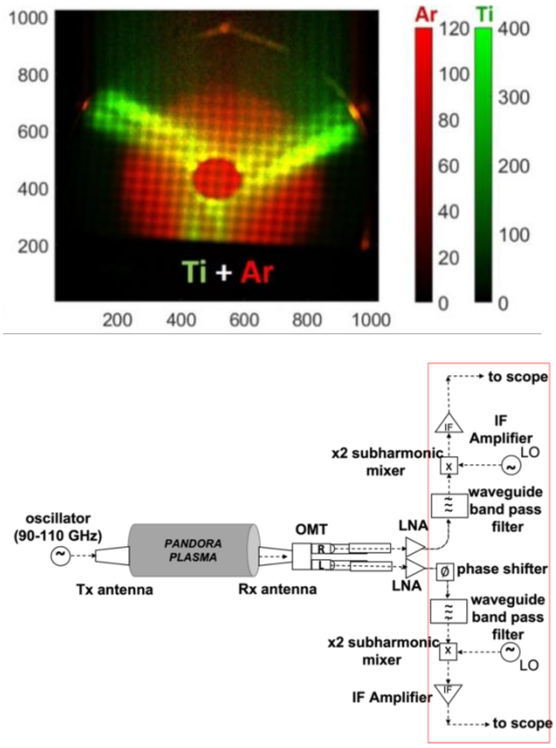


Figure 1: Top - Typical X-ray energy-filtered image where it is possible to distinguish fluorescence X-rays coming from plasma (Ar) and from chamber walls materials (Ti). Bottom - Block-diagram of the mm-wave polarimeter VESPRI 2.0.

References:

1. D. Mascali et al. (2022) Universe 8, 80.
2. E. Naselli et al., JINST 17 (2022) C01009.
3. S. Biri et al., JINST 16 (2021) P03003.
4. E. Naselli et al., Cond. Matter 7(1) (2022) 5.
5. G. Torrisi et al. (2022) Front. Astron. Space Sci. 9 949920.

Plasma Generation and Expansion in the Vacuum Arc Remelting (VAR) process

E. Karimi-Sibaki [1,2,*], A. Kharicha [1,2]
[1] Christian-Doppler Laboratory for Metallurgical Applications of Magnetohydrodynamics,
Montanuniversitaet of Leoben, Franz-Josef-Str. 18, 8700, Leoben, Austria
[2] Chair of Simulation and Modeling of Metallurgical Processes,
Montanuniversitaet of Leoben, Franz-Josef-Str. 18, 8700, Leoben, Austria

Abstract:

The Vacuum Arc Remelting (VAR) process plays a pivotal role in the purification of a wide range of alloys, including stainless steel, Nickel-based alloys, and Titanium-based alloys. This advanced technique entails heating the electrode's tip (impure alloy) through cathode spots, generating a DC plasma arc in a vacuum environment. The melting of the electrode tip forms molten droplets that move through the vacuum into a molten pool. The pool then solidifies within a water-cooled mold, providing exceptionally high-grade, ultra-clean alloys [1].

The plasma arc is maintained between the tip of the melting electrode (cathode) and the top of the ingot (anode). The existence of the arc depends on cathode spots that exhibit erratic movement across the cathode's surface. The arc, which emanates from these spots, consists of ions and electrons carrying a substantial electric current ranging from 5 to 40 kA. This arc plays a crucial role in the overall energy transfer to the top of the ingot, influences the flow within the molten pool, and, as a result, significantly impacts the quality of the final ingot. Therefore, predicting the behavior of cathode spots and arc plasma is of utmost importance.

During the operation of the VAR process, we recorded the behaviors of cathode spots and plasma arc using a high-speed camera (Phantom v2512) that captured 60,000 frames per second. We discovered that the spots tend to initiate and persist within the partially melted surface at the center of the electrode tip. Any existing spots outside the melting zone accelerate toward the electrode's edge, leading to their extinguishment. Throughout the operation, we observed a relatively symmetrical and central plasma column [2].

Cathode spot dynamics was numerically modeled, assuming a combination of stochastic random motion and drift in the retrograde direction [3]. The movements of a cluster of spots, i.e. swinging or rotating, were captured. Two-fluid magnetohydrodynamics (MHD) model of plasma to investigate the arc column as a mixture of two continuous interpenetrating compressible fluids involving ions and electrons was employed [2]. Calculated plasma parameters, such as the number density of ions/electrons, electric current density, flow of ions/electrons, and temperature of ions/electrons, were thoroughly analyzed. Numerical results were verified considering the light intensity for the observed arc column in our experiment. Examples of results are shown in Figure 1. This comprehensive study offers valuable insights into the generation and expansion of plasma in the VAR process.

Keywords: Vacuum arc remelting, DC arc, cathode spots, plasma generation and expansion, high-speed camera, modeling and numerical simulations, plasma processing of alloys

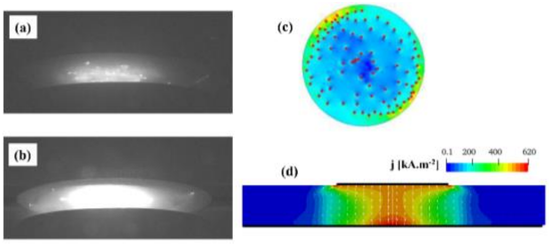


Figure 1: Experimental (a) cathode spots and (b) arc plasma. Calculated (c) instantaneous spot's distribution on electrode tip, (d) arc current density in the vacuum.

References:

1. Karimi-Sibaki, E., Kharicha, A., Wu, M. et al. (2020) A Parametric Study of the Vacuum Arc Remelting (VAR) Process: Effects of Arc Radius, Side-Arcing, and Gas Cooling. *Metall Mater Trans B* 51, 222–235
2. Karimi-Sibaki, E., Peyha, M., et al. (2022) Experimental and numerical investigations of arc plasma expansion in an industrial vacuum arc remelting (VAR) process. *Sci Rep* 12, 20405.
3. Kharicha, A., Karimi-Sibaki E., et al. (2018) Numerical Investigation of Collective Motion of Cathode Spots. *28th Int. Symp. Discharges and Electrical Insulation in Vacuum (ISDEIV)*

Simulation of an ion beam extraction from an ICP by using a self-consistent plasma sheath model.

K. M. Rettig [1,2,*], T. Dunger[1], E. Loos[1], M. Nestler[1], J. Schuster[2]
[1] scia Systems GmbH, Chemnitz, Germany
[2] Fraunhofer Institute for Electronic Nano Systems ENAS, Chemnitz, Germany

Abstract:

Ion beam processing of substrate surfaces requires customized beam characteristics depending on the target application. For example, the beam width must be controlled in ion beam trimming (IBT), where a focused ion beam locally corrects topography errors of films or structures.

We present a simulation approach which aims to predict properties of the inductively coupled plasma (ICP) and extracted ions. The simulation model also enables, investigations of process optimizations and digital prototyping of novel ion beam source concepts.

Additionally, we characterized an Argon ion beam extracted from a 37 mm RF ion source through a three-grid accelerator-system experimentally, to validate the theoretical model.

Ion beam simulation approaches typically require knowledge of the plasma parameters present in the discharge chamber. A global model for gridded ICP sources [1] has been implemented, to overcome the difficulties of diagnostic measurements in an industrial type ion source. The model calculates plasma properties based on the chamber geometry and applied operation conditions. The resulting plasma parameters are provided as initial conditions to a numerical beam extraction simulation. Thereby, the particle movement through the extraction geometry is calculated using a particle-in-cell (PIC) and direct-simulation Monte-Carlo (DSMC) approach [2]. The model yields a simulated ion beam, that matches with the experimental data on beam width and extracted ion current, both qualitatively and quantitatively.

An important factor for a realistic ion beam extraction simulation, is the formation of the plasma sheath over each extraction aperture. This is represented by an extraction-grid transparency-factor for ions in the global model. The factor affects the balance between plasma density and extracted ion current and is estimated from beam measurements. To be independent of experimental investigations, we developed a self-consistent model that iteratively calculates the sheath properties from the plasma parameters given by the global model. Therefore, the sheath is considered to be a sharp boundary between plasma and extracted beam at a field strength of $|E_\mathrm{P}| = T_\mathrm{e}/\lambda_\mathrm{D}$ [3]. The resulting transparency-factor is then used to calculate the plasmas properties within the next iteration. This process repeats until convergence is reached. A comparison of the results from the iterative scheme and the use of experimental input shows good agreement, as depicted in Figure 1. Thus, the model can be applied to simulate an ion beam extraction self-consistently, by only providing the source geometry and operating conditions.

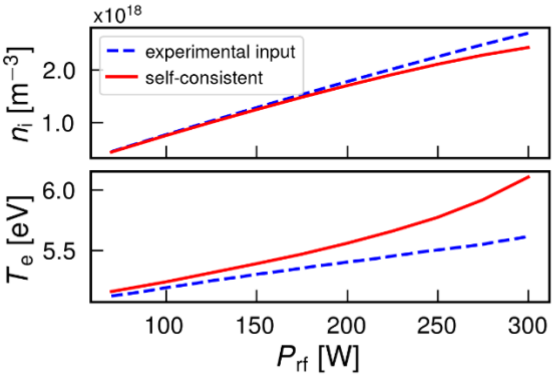


Figure 1: Comparison of the calculated ion density n_i and electron temperature T_e from the global model for ICP with experimental input (blue) and self-consistent (red) over a range of applied RF powers.

Keywords: inductively coupled plasma (ICP), ion beam extraction, gridded ion source, focused broad ion beam, plasma simulation, particle-in-cell, global plasma model.

References:

1. Chabert, P. et al. Global model of a gridded-ion thruster powered by a radiofrequency inductive coil. Physics of Plasmas, **19**(7), 073512 (2012).
2. Fasoulas, S. et al. Combining particle-in-cell and direct simulation Monte Carlo for the simulation of reactive plasma flows. Physics of Fluids, **31**(7), 072006 (2019).
3. Kovaleski, S. D. Calculation of the ion extraction boundary of a plasma ion source, IEEE Transactions on Plasma Science, **34**(1), 23-27 (2006).

Application of high power ns-pulsed plasma for methane reforming

M. Gromov[1], Georgios D. Stefanidis[2,3], R. Morent[1], Anton Nikiforov[1]
[1] Department of Applied Physics, Ghent University, Research Unit Plasma Technology
Sint-Pietersnieuwstraat 41, 9000, Ghent, Belgium,
[2] School of Chemical Engineering, National Technical University of Athens, Iroon Polytechniou 9, 15780, Athens, Greece
[3] Laboratory for Chemical Technology, Ghent University, Tech Lane Ghent Science Park 125, B-9052, Gent, Belgium

Abstract:
At present chemical industry is almost exclusively based on the use of fossil fuels leading to enormous CO_2 emission above 900 Mt a year with an expected increase of 8% annually. Methane reforming is one of the most energy-demanding and CO_2 emitting processes where decarbonization of the reforming process can provide a sustainable way of reaching the goal of EU for 2050 on the reduction of fossil fuels and chemical industry electrification. On the way to achieve this challenging goal, one of the possible pathways is the use of plasma from electrical discharges. The advantages of plasma-initiated methane reforming are: applicability at a low scale; use of electricity for gas heating instead of fossil fuel-based heating; flexibility of the process and coupling with renewable electricity production; zero CO_2 footprint. Despite obvious advantages, the plasma-based CH_4 reforming was not yet implemented on an industrial scale as a deep understanding of the fundamental processes behind the reforming is required at first.

In the current project, we investigated the production of valuable hydrocarbons from CH_4/H_2 mixture in high-power pulsed discharge at variable operational pressure. The discharge was generated by 10 ns pulses with at least 2 MW pulsed power in a pin-to-plate electrode arrangement, Figure 1. The pulse discharge generation process was studied by emission spectroscopy, plasma imaging and laser scattering. Gas ionization in conditions of high-power pulses was investigated through time-resolved Stark broadening analysis of H_α line. Gas heating kinetics during the pulse was analyzed based on the detection of C_2 Swan band appearing in the discharge spectra during the first 500 ns whereas gas temperature in the after-pulse phase was investigated by detection of Rayleigh laser scattering.

It was found that the discharge constriction strongly depends on the gas pressure. The plasma filament of 450 µm diameter was formed at 500 mbar that decreased till 150 µm at 1 bar which then stayed almost constant till the highest tested pressure of 3 bar. Due to the high energy injected per pulse, the ionization degree in the discharge core could reach 0.5% or electron density of 1.8×10^{23} m^{-3} at 3 bar pressure and strongly depended on the pressure.

Importantly, gas heating kinetics showed that extensive gas heating was taking place during the discharge. The gas temperature rose during the discharge phase and after the pulse on µs time scale led to the initiation of thermal CH_4 reforming. The heating kinetics was analyzed based on C_2 emission and Rayleigh scattering and confirmed the negligible contribution of non-thermal processes in CH_4 reforming in conditions of pulsed high-power electrical discharges operating at a pressure range of 500 mbar - 3 bar.

Keywords: methane reforming, pulsed discharge, emission spectroscopy, gas heating, chemical process electrification.

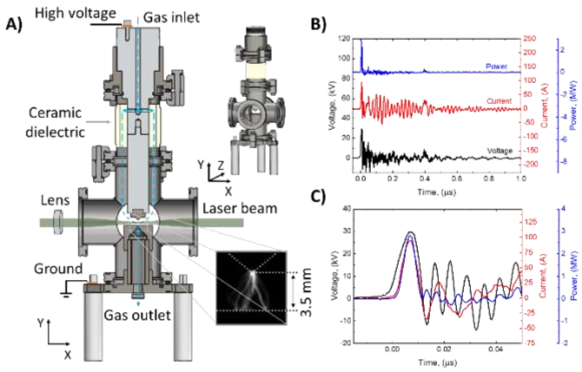


Figure 1: Experimental set-up: (A) Schematic drawing of the ns-pulsed discharge used for CH_4 reforming; (B) 1 µs resolved voltage, current and power pulse waveform; (C) high resolution waveform of voltage, current and power of the discharge

Acknowledgements:

This work was performed in the framework of the Moonshot clusterSBO project P2O ("Power-to-241 olefins: electrified steam cracking and plasma booster"), supported by VLAIO Agency.

On the role of reaction mechanisms during the plasma-assisted dry reforming of methane

M. Oliva-Ramírez [1,2,*], P. Navascués [1], J. Cotrino [1,2], A. R. González-Elipe [1], A. Gómez-Ramírez [1,2]

[1] Laboratory of Nanotechnology on Surfaces and Plasma, Instituto de Ciencia de Materiales de Sevilla (CSIC-Universidad de Sevilla), Seville, Spain
[2] Departamento de Física Atómica, Molecular y Nuclear, Universidad de Sevilla, Seville, Spain

Abstract:

Plasma reactions of CO_2 + CH_4 mixtures have been proposed as a suitable process for the dry reforming of methane. Without specific catalysts, most studies report the formation of CO and H_2 as main reaction products and arise the question whether CH_X radicals coming from CH_4 may interact with intermediate species formed by electron impact dissociation of CO_2, a critical step for the formation of high added value oxygenated compounds. We have addressed this question studying the CO_2 + CH_4 plasma reaction in a ferroelectric-moderated packed-bed reactor varying the reactants ratio. Analysis of the reaction products by mass spectrometry and the plasma reaction intermediates by optical emission spectroscopy suggest that few direct cross-link interactions exist between intermediate plasma species issued from CH_4 or CO_2. This preliminary evidence is corroborated by experiments using $^{13}CO_2$ instead $^{12}CO_2$ as reactant. The isotope labeling procedure has proved that plasma reaction mechanisms of CO_2 and CH_4 molecules proceed almost independently, with the formation of small amounts of water and the removal of carbon deposits resulting CH_4 plasma decomposition as sole evidences of cross reactions. These results highlight the need of using catalysts to promote specific surface reactions for a better control of the selectivity of the process.

Keywords: Methane dry reforming, Hydrogen production, Packed-bed plasma reactor, Isotope labeling, Plasma-catalysis, Plasma-assisted processes

References:

1. Navascués, P., Cotrino, J., González-Elipe, A.R., Gómez-Ramírez, A. (2023) Plasma assisted dry reforming of methane: Syngas and hydrocarbons formation mechanisms, *Fuel Process. Technol.*, 248, 107827.

Silicon Carbide for neutron diagnostic in plasma

S. De Luca [1], C. Altana [1], S. Amaducci [1], L. Cosentino [1], S. Tudisco [1]
[1] Istituto Nazionale di Fisica Nucleare, Laboratori Nazionali del Sud, Catania, Italy

Abstract:
The exploitation of fusion technology demands the development of a new generation of plasma diagnostic, capable to operate in the reactors harsh environment. Neutron monitors based on solid state silicon detectors is well established for neutron beams [1] and monitoring or nuclear waste [2]. Even if standard silicons are unable to face the condition of reactors such ITER, the aforementioned requirements are met by Silicon Carbide (SiC) detectors. A new generation of SiC (figure 1) has been developed at INFN-LNS [3], with high resistance to radiation, fast response time, stability and good energy resolution. Such devices, thanks to their capability to operate in harsh environment, are really promising not only for plasma diagnostics in fusion reactors, but also for nuclear physics with high intensity beams and in laser facility. We will present the extended characterization of this devices, focused on the aspect of resistance to neutron irradiation from thermal energy to tens MeV. In particular, a campaign of irradiation was performed at the n_TOF facility, where exposed to an integrated neutron fluence of 10^{14} neutrons per cm^2, peaked in the MeV energy region, comparable with the values reached inside a fusion reactor.

Keywords: neutron, silicon, silicon carbide, plasma diagnostic, radiation hard.

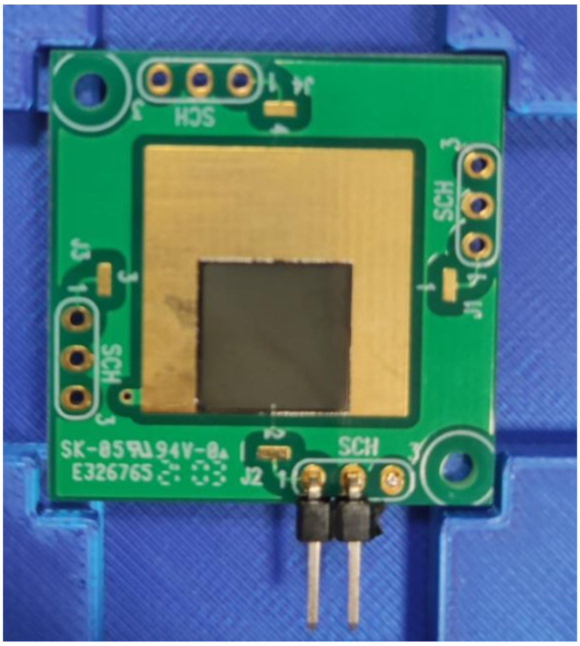


Figure 1: Image of a SiC detector.

References:

1. Cosentino, L., et al. (2015) Silicon detectors for monitoring neutron beams in n-TOF beamlines, *Control.Rev. Sci. Instrum. 86, 073509.*
2. Cosentino, L. et al. (2021), SiLiF Neutron Counters to Monitor Nuclear Materials in the MICADO Project, *Sensors*, 21(8), 2630.
3. Tudisco, S., La Via, F., Agodi, C., Altana, C., Borghi, G., Boscardin, M., ... & Zimbone, M. (2018). SiCILIA-Silicon Carbide detectors for Intense Luminosity Investigations and Applications. *Sensors, 18(7), 2289*.

A New Generation of Microreactor for Gas/Liquid Plasma Chemical Processes : Study of Segmented Flow under Plasma

P. Dedieu [1], G. Morand [1], S. Ognier [1] and M. Tatoulian [1]
[1] Institut de Recherche de Chimie de Paris, PSL Université, Paris, France

Abstract:

The development of gas/liquid plasma chemical processes is hindered by the difficulty to control the gas/liquid interface under plasma conditions. Microreactors have recently received attention due to their ability to stabilize biphasic interfaces while allowing a low inter-electrode distance and enhanced mass and heat transfers [1]. Biphasic gas/liquid segmented flow in particular represents best compromise between maximizing the gas/liquid interface area while using gas to liquid flow rates conducive to chemical processes [2]. However, segmented flow use for plasma processes have barely been studied. We report the development of a new generation of microreactor for gas/liquid plasma chemical processes (Figure 1). The study of segmented flows generated with argon and fifteen different liquids commonly used in chemical synthesis shows the necessity to integrate liquid vapour pressure to literature scaling laws to effectively predict bubble and slug lengths, and bubble residence time. Plasma generation was performed in segmented flows using all liquids and changes the flow pattern because of gas heating it causes. Based on liquid boiling point and dielectric constant, we designed a flow map which makes it possible to predict how plasma will modify the flow pattern. Plasma power and specific energy input depend on liquid to gas ratio, which makes fluidic conditions tunable to plasma parameters requirements. Bubble lengthening due to gas heating by plasma greatly impacts bubble residence time in the reactor and can be correlated to specific energy input. Both results highlight the influence of liquid phase on plasma generation. A compromise in fluidic conditions therefore has to be found to maximize both residence time and plasma power. This new generation of microreactor is further used in gas/liquid plasma assisted cyanation of aromatics by acetonitrile, a challenging reaction in developping greener and safer synthetic routes towards nitriles. Conversions of up to 60 % with selectivities in cyanated compounds of 70-80 % were achieved, which highlights the possible applications of such microreactors in gas/liquid plasma chemical processes.

Keywords: gas/liquid plasma, microreactor, segmented flow, plasma chemical processes

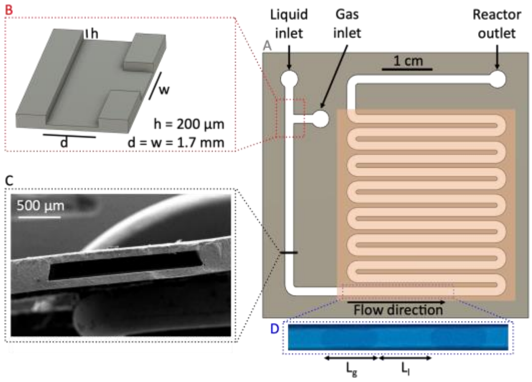


Figure 1: Illustration of the microchannel used to generate plasma in a gas/liquid segmented flow (A). The dimensions of the T-junction where the flow is generated (B), a scanning electron microscopy cut view of the channel (C) and a picture of an argon/ethanol segmented flow typically observed (D) are shown. The orange rectangle stands for the electrode and shows its shape and position.

References:

1. J. Wengler, S. Ognier, M. Zhang, E. Levernier, C. Guyon, C. Olivier, L. Fensterbank and M. Tatoulian, *Reaction Chemistry & Engineering*n 2018, **3**, 930-941
2. M. T. Kreutzer, F. Kapteijn, J. A. Moulijn and J. J. Heiszwolf, *Chemical Engineering Science*, 2005, **60**, 5895-5916

Plasma-catalytic hybrid process for CO_2 valorization into liquid fuels

Noelia Merino[1,3], Stéphanie Ognier[1,*], Xavier Duten[2], Maria Mikhail[3], Michael Tatoulian[1]
[1]Institut de Recherche de Chimie Paris, UMR 8247 (CNRS – Chimie ParisTech),
Equipe 2PM, Paris, France
[2]LSPM – CNRS UPR 3407 – Université Sorbonne Paris Nord, Villetaneuse, France
[3]ENERGO, Hellemmes – Lille, France
* stephanie.ognier@chimieparistech.psl.eu (corresponding author)

Abstract:

Greenhouse gases such as CO_2 and CH_4 are one of the main causes of climate change. Efforts are being made to reduce their emissions as well as to valorize them. As current thermal-catalysis processes used for the valorization of CO_2 require high temperatures and pressures, research is conducted to find a more direct approach to convert CO_2 and CH_4 into higher value-added liquid products under mild conditions. Non-thermal plasmas are very appealing because they carry highly energetic species that can cause excitation, ionization or even dissociation of gas molecules, while remaining at atmospheric pressure and relatively low temperatures. Combining plasma and heterogeneous catalysis could enable to produce selectively liquid products in mild conditions thanks to the unique synergies between plasma and catalysis.

Several studies have already been conducted with plasma-catalysis for the conversion of CO_2 and CH_4 into liquid products, most frequently using a dielectric barrier discharge (DBD) reactor[1,2,3,4]. They show promising results, with conversions up to 38% for CO_2 and 50% for CH_4 and a selectivity in liquid products up to 48%[1].

In this study, the experimental set-up consists in a catalytic dielectric barrier discharge (DBD) reactor. The catalysts used are metal oxide based. This work explores how CO_2 and CH_4 conversion and the selectivity into liquid products are influenced by experimental parameters such as temperature, electric power, frequency, ratio CO_2/CH_4 and gas hourly space velocity (GHSV) and by physico-chemical properties of the catalyst. The findings of this study can be used to optimize the conditions required to obtain the desired products, and to understand the synergistic effects of plasma-catalyst interactions.

Keywords: plasma-catalysis, non-thermal plasma, CO_2 valorization, liquid fuels, process engineering

References:

1. Li, D., Rohani, V., Fabry, F., Ramaswamy, A. P., Sennour, M., & Fulcheri, L. (2020). *Direct conversion of CO2 and CH4 into liquid chemicals by plasma-catalysis.* Applied Catalysis B: Environmental, 261, 118228.
2. Li, J., Dou, L., Gao, Y., Hei, X., Yu, F., & Shao, T. (2021). *Revealing the active sites of the structured Ni-based catalysts for one-step CO2/CH4 conversion into oxygenates by plasma-catalysis.* Journal of CO2 Utilization, 52, 101675.
3. Dou, L., Liu, Y., Gao, Y., Li, J., Hu, X., Zhang, S., ... & Shao, T. (2022). *Disentangling metallic cobalt sites and oxygen vacancy effects in synergistic plasma-catalytic CO2/CH4 conversion into oxygenates.* Applied Catalysis B: Environmental, 318, 121830.
4. Wang, Y., Fan, L., Xu, H., Du, X., Xiao, H., Qian, J., ... & Wang, L. (2022*). Insight into the synthesis of alcohols and acids in plasma-driven conversion of CO2 and CH4 over copper-based catalysts.* Applied Catalysis B: Environmental, 315, 121583.

Degradation of organic pollutants in water through plasma – catalysis process

D. Tsokanas[1], O. Dolinski[1], S. Meropoulis[1], C. Aggelopoulos [1*]

[1] Institute of Chemical Engineering Sciences, Foundation for Research and Technology Hellas (FORTH/ICE-HT), Patras, Greece

Abstract:
Water pollution is considered one of the main environmental challenges that must be effectively addressed. The presence of organic pollutants in wastewater is one of the main causes of water pollution, with serious negative effects on human health and the environment (1). Therefore, developing more environmentally friendly and sustainable technologies for the degradation of these organic pollutants is a one-way road. Among the numerous methods, advanced oxidation processes (AOPs), which base their action on the generation of nonselective and strong oxidizing species (e.g. ·OH, O_3, etc.), are considered advantageous compared to conventional physicochemical and biological processes (2).
Cold atmospheric plasma (CAP) is regarded as a novel and very promising AOP for the rapid and highly efficient destruction of persistent organic pollutants in environmental matrices and when combined with catalysis the pollutant degradation and energy efficiency can be further improved (plasma-catalysis) (3). In the present study, TiO_2 and/or ZnO were combined with underwater plasma microbubbles (PMB), energized by low-frequency high voltage nanopulses (Figure 1), and compared under the same experimental conditions for the degradation of various organic pollutants, such as dyes and antibiotics. The effect of several critical parameters on pollutant degradation and mineralization was investigated (treatment time, pulse voltage, plasma gas, catalyst loading), while the plasma-catalytic mechanism was thoroughly explored. The present study can contribute to providing fundamental insights and further expand of plasma-catalysis for water treatment.

Keywords: Plasmacatalysis, Plasma bubbles Dielectric barrier discharge, TiO_2, ZnO, Wastewater treatment

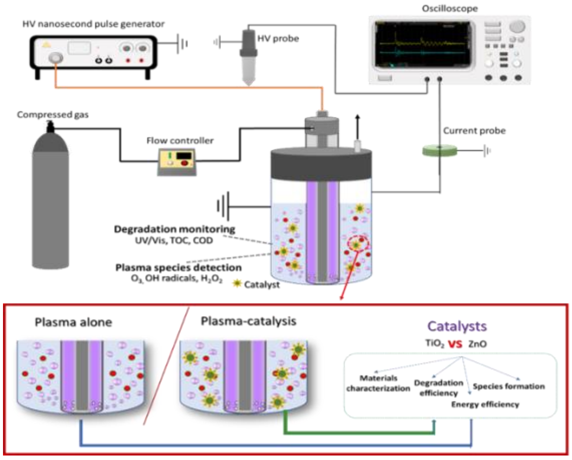


Figure 1: Schematic representation of the experimental setup used to treat the pollutant by plasma bubbles in the absence and presence of TiO_2 and ZnO and characterize the plasma**.**

References:

1. Mukhopadhyay, A., Duttagupta, S., Mukherjee, A. (2022) Emerging organic contaminants in global community drinking water sources and supply: a review of occurrence, processes and *remediation J. Environ. Chem. Eng.*, 10, 107560.
2. Kumar, V., Shah, M.P. (2021) (Ed.), Advanced Oxidation Processes for Effluent Treatment Plants, Elsevier, pp. 1-31.
3. Aggelopoulos C.A. (2022) Recent advances of cold plasma technology for water and soil remediation: a critical review, *Chem. Eng. J.*, 428 (2022), 131657.

Acknowledgements:

H.F.R.I. Hellenic Foundation for Research & Innovation

The research project was supported by the Hellenic Foundation for Research and Innovation (H.F.R.I.) under the "2nd Call for H.F.R.I. Research Projects to support Faculty Members & Researchers" (Project Number: 3560).

Electron emission Electron Emission Yield of Cu, and Au: Effects of Electron Irradiation and Argon Etching

M. Belhaj [1,*], S. Dadouch [1]
[1] ONERA, DPHY, Université de Toulouse, 31055

Abstract: The electron emission induced by electron impact plays a crucial role in various applications, notably in multipactor phenomena and e-cloud effects in particle accelerators. It has been established that prolonged irradiation of metal surfaces leads to a reduction in their electron emission capability, a phenomenon commonly referred to as conditioning. Several hypotheses have been proposed to explain this phenomenon. Some attribute it to a change in the type of oxide formed on the surface, while others consider the transformation of hydrocarbon contamination from sp3 to sp2 hybridization as the main cause.

In this context, we decided to examine the effect of conditioning with a 500 eV electron beam on the surfaces of different metals, combining in situ measurements of electron emission yield with surface analysis by X-ray photoelectron spectroscopy (XPS).

Our study focuses on both technical surfaces (exposed to ambient air) and surfaces purified in situ by ionic erosion using an argon ion source. Our comparative results, obtained on a surface prone to oxidation (copper) and a surface considered non-oxidizable (gold), indicate that hydrocarbon contamination appears to play a predominant role in the conditioning process, in both cases.

Keywords: Secondary electron, electron emission yield, conditioning. Agon etching

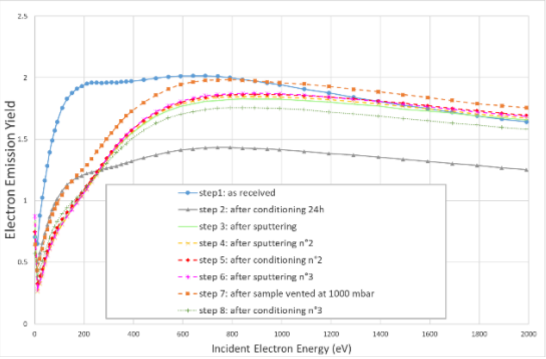


Figure 1: Electron emission yield variation as function of the surface processing.

Plasma surface treatment of aluminium sheets

P. I.Kovács [1,2,*], M. Berczeli [1] Z. Weltsch [2]
[1] Department of innovative vehicles and materials, John von Neumann University, Kecskemét,Hungary
[2] Department of Road and Rail Vehicles, Zalazone Innovation park, Széchenyi István University, Győr, Hungary

Abstract:

Weight reduction is an important issue in all engineering fields. This includes the ever-evolving automotive industry, where weight reduction is essential in a variety of applications. One reason for this is that European Union regulations are imposing ever stricter emission requirements on manufacturers every year. There are several engineering solutions with different approaches to achieve pollution reduction. One suitable way to reduce the weight of vehicle bodies is to use modern material combinations. The engineering use of these materials may be limited by the requirement for different installations. Thus, in addition to the development of materials, particular attention must be paid to their bonding technology. For example, when using aluminum sheets, the surface condition of the different materials can be a problem. Adhesive bonding may therefore be an optimal technology for joining identical or dissimilar materials. Different preparation may be required to bond surface elements. My experiments aim to investigate the physical, topographical, and chemical characteristics of the surfaces. This will help me to explore the material science context, how I can influence the quality of the adhesion bonding on surfaces. The aim of my research is not only to modify the surface physics and chemistry of the raw materials from the production process, but also to modify these properties in a conscious and engineering-oriented way. I want to fill a scientific gap in the international literature on the relationship between surface modification of raw materials and bonding technology. How specific physical and chemical processes take place, their duration of action and their role in bonding technologies.

Keywords: surface treatment, ambient pressure plasma, adhesive technology, surface wetting.

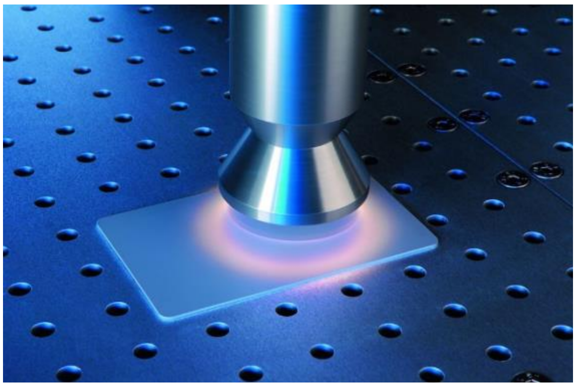


Figure 1: Surface activation with atmospheric pressure plasma.

References:

1. https://www.plasmatreat.com/en/what-is-plasma/plasma-surface-treatment/surface-activation/

SICT 2024 / Tribology 2024 Joint Session I. C: Surface Engineering, Coatings and Tribology

The Astonishing Diversity in the World of 2D Materials - Lessons learned from MXenes and Transition Metal Carbo Chalcogenides (TMCCs) for Solid Lubrication

C. Gachot
Technische Universität Wien, Lehárgasse 6, 1060 Vienna

Abstract:

Tribology, the science of friction and wear, is gaining more public attention as an efficient, reliable, and sustainable operation of machine elements is becoming increasingly important. This is relevant for almost all industrial sectors since machines and mechanical systems with numerous moving parts being in relative motion are vital for most systems [1]. By estimation of Holmberg and Erdemir, 23% of the world's energy consumption originate from tribological contacts [1]. Lubricants are commonly introduced between the rubbing surfaces, thus minimizing friction and wear. The most common lubricants are petroleum-based mineral oils in liquid form [2]. However, diminishing oil resources, the need for ever lower frictional losses, as well as higher demands on the lubricants in terms of resistance against extreme conditions such as high temperatures or low environmental pressures push liquid lubricants to their limits [3]. It is expected that new lubricant technologies will contribute greatly to a reduction of friction and wear such as the use of new 2D materials in particular MXenes, Transition Metal Carbo Chalcogenides (TMCCs) or Black Phosphorous (BP). In this talk, examples for new 2D materials will be given and discussed under the light of energy savings and sustainability.

References:

1. Holmberg, K., & Erdemir, A. (2017). Influence of tribology on global energy consumption, costs and emissions. Friction, 5(3), 263–284. doi: 10.1007/s40544-017-0183-5
2. Shahnazar, S., Bagheri, S., & Abd Hamid, S. B. (2016). Enhancing lubricant properties by nanoparticle additives. *International Journal of Hydrogen Energy*, *41*(4), 3153–3170. doi: 10.1016/j.ijhydene.2015.12.040
3. Priest, M., & Taylor, C. M. (2000). Automobile engine tribology—approaching the surface. *Wear, 241*(2), 193–203. doi: http://dx.doi.org/10.1016/S0043-1648(00)00375-6

The effect of ZrO_2 nanoparticles reinforcement and heat treatment on tribological properties of electroless Ni-P coatings with high P content

E. Baroni [1,*], G. Pedrizzetti [2], G. Pulci [2], F. Marra [2], M. Merlin [1]
[1] Department of Engineering (DE), University of Ferrara, Ferrara, Italy
[2] Department of Chemical Engineering, Materials, Environment, Sapienza University of Rome, INSTM Reference Laboratory for Engineering of Surface Treatments, Rome, Italy

Abstract:

Electroless Ni-P coatings have been widely used in recent years for wear and corrosion protection of industrial steel components [1]. In particular, coatings with high P content (HP) have been developed for the energy production industry to guarantee optimized corrosion resistance in aggressive environments. The most adopted strategies to confer good wear resistance to the coatings and enhance their durability are the addition of functional particles and post-fabrication heat treatments [2]. In the literature, the understanding of the tribological behavior of Ni-P coatings is not yet completely understood, specifically as concerns the wear mechanisms and the effect of different counter-body materials [3].

In the present work, both the single and combined effect of the addiction of ZrO_2 nanoparticles and heat treatment with different temperature-time schedules on the tribological resistance of HP coatings was investigated. Dry wear tests were conducted in a ball-on-disk configuration using a reciprocating tribometer. Disks in carbon steel were coated by Ni-P, part of them reinforced with ZrO_2 nanoparticles and/or subjected to heat treatment. Two different temperatures (340 °C and 400 °C) and dwell times between 0.5 h and 8 h were selected as heat treatment parameters. As preliminary analyses, microhardness and scratch tests were performed on the disks, demonstrating that the most promising properties could be obtained for nanocomposite coatings after thermal treatments at 340°C for 8 h and 400°C for 1h.

Wear studies were then performed on bare HP coatings, standard nanocomposite and nanocomposite annealed with the above-mentioned temperature-time schedules. Balls in100Cr6and Al_2O_3 with a diameter equal to 6 mm were used as counterbody materials.

Reciprocating sliding wear tests were carried on at a constant reciprocating frequency equal to 2 Hz, with a 10 mm displacement to reach a total sliding distance of 100 m under normal loads equal to 10 and 30 N. Three replicas were performed for each tested condition. The coefficient of friction (COF) and overall system wear were continuously measured during the tribological tests. Ball wear scars were analyzed by stereomicroscopy, while wear scars in charge of disks were measured by a 3-D optical non-contact profilometer to determine the volume loss. The specific wear rate (WR) was calculated both for balls and disks. Worn surfaces were also analyzed through scanning electron microscopy (SEM) to identify the main wear mechanism according to the different test conditions.

The main results showed that, depending on the counterbody material, NiP has a different tendency to oxidation. COF and WR resulted also in being differently influenced by tribo-oxidation as main wear mechanism in the different tested conditions. All NiP-ZrO_2 nanocomposite coatings showed different tribological behavior depending on the applied load. Ni grain size and Ni_3P precipitation and coarsening determined by the two different heat treatment were found having an influence on their wear properties.

Keywords: nanocomposite Ni-P coatings, ZrO_2 reinforcement, heat treatment, reciprocating sliding, friction, wear, microstructural analysis

References:

1. Krishnan, K. H et al., (2006), An Overall Aspect of Electroless Ni-P Depositions - A Review Article, *Metall Mater Trans A*, 37A, 1917-1926.
2. León-Patiño, C.A. et al., (2019), Tribological characterization of heat-treated Ni-P and Ni-P-Al2O3 composite coatings by reciprocating sliding tests, *Wear*, 426-427, 330-340.
3. Aslanyan, I.R. et al., (2006), Effect of reinforcing submicron SiC particles on the wear of electrolytic NiP coatings - Part 1. Uni-directional sliding, *Surf Coat Technol*, 201, 581-589.

A Comparative Study on the Effect of Roughness Parameters on the Adhesion and Tribological Properties of PEEK Coatings Deposited on Mild Steel Substrates

Amal Ameen Seenath[1], Abdul Samad Mohammed[1,2,*]
[1] Mechanical Engineering Department, King Fahd University of Petroleum & Minerals, Dhahran – 31261, KSA
[2] Interdisciplinary Research Center of Advanced Materials, King Fahd University of Petroleum & Minerals, Dhahran – 31261, KSA
*Corresponding Author: samad@kfupm.edu.sa

Abstract:

Polymer coatings play a crucial role in various industries, providing protection and enhancing the performance of materials. One important aspect in a coating system is the surface roughness parameters of the substrate, which greatly influence their adhesion properties, which in turn affects the coating tribological performance. The adhesion of a polymer coating is the strength with which it bonds to the underlying material. A strong adhesion ensures that the coating remains intact and firmly attached to the substrate, even under harsh conditions. Understanding the relationship between surface roughness and adhesion is vital for optimizing the performance of polymer coatings.

Surface roughness refers to the irregularities or deviations present on the surface of a material. These irregularities can range from microscopic to macroscopic scales and can greatly impact the adhesion of the coating to the substrate. Surface roughness parameters, such as average roughness (Ra), root mean square roughness (Rq), peak-to-valley height (Rz), the kurtosis and the skewness of the peak and valley distributions directly affect the adhesion strength of the coating.

The average roughness (Ra) is a commonly used parameter to quantify the surface roughness of a material. It represents the arithmetic mean of the absolute values of the surface height deviations from the mean line. A higher Ra value indicates a rougher surface, which can enhance the adhesion of the polymer coating. The increased surface area resulting from higher roughness provides more contact points for the coating to adhere to, improving its overall adhesion strength.

Root mean square roughness (Rq) is another important parameter that characterizes the surface roughness. It represents the square root of the average of the sum of the squared height deviations from the mean line. Rq provides a more comprehensive measure of the roughness, considering both the height and distribution of the irregularities. A higher Rq value signifies a more uneven surface, which can positively impact the adhesion of the polymer coating.

Peak-to-valley height (Rz) measures the vertical distance between the highest peak and the lowest valley within a sampling length. It provides information about the extreme irregularities present on the surface. A higher Rz value indicates a greater variation in surface height, which can enhance the mechanical interlocking between the coating and the substrate, resulting in improved adhesion.

Kurtosis measures the sharpness of the surface peaks and valleys, skewness reflects the asymmetry of the profile, which can enhance the adhesion strength of the coatings.

Hence, optimizing the surface roughness parameters of polymer coatings is crucial for achieving the desired adhesion strength. However, it is important to note that there is no universal roughness value that guarantees optimal adhesion. The ideal roughness parameters depend on various factors, including the type of coating, substrate material, and intended application.

Thus, the main focus of the current study is to evaluate the effect of these roughness parameters on the adhesive strength and the tribological performance of PEEK polymer coatings deposited on a mild steel substrate by an electrostatic spraying technique. Two substrates with varying roughness parameters as shown in Table 1, were used to deposit a 45±5 µm thick

PEEK coatings, and their adhesion strength and tribological performance are evaluated using a ball-on-disk wear test with a 6.3 mm diameter alumina ball as a counterface. Wear tests were conducted at room temperature with varying normal loads (30. 40 and 50 N) at a constant speed of 0.2 m/s for 10,000 cycles. A 3D optical profilometer (GTK-A, Bruker, USA) was used to determine the different surface parameters, namely: maximum peak-to-valley distance (R_t)-height parameter, the peak density measurement, and other roughness parameters including skewness and kurtosis as shown in Table 1. The study of the morphology and wear mechanism was conducted by scanning electron microscopy (SEM).

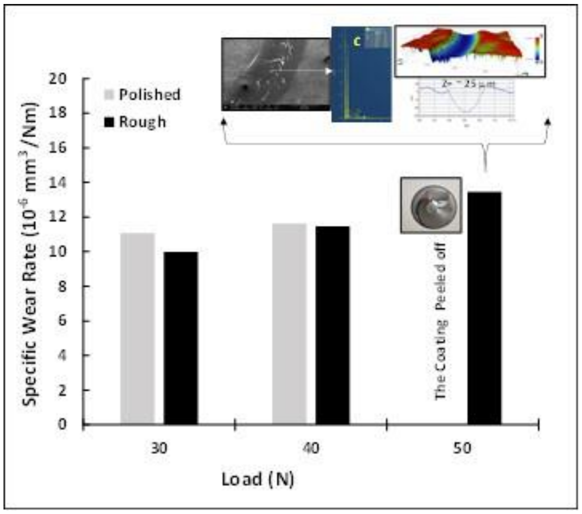


Figure 1. Variation of Specific wear rate and corresponding SEM, EDS spectra, and 3D wear profile of the rough sample at a load of 50 N

The water contact angle measurements were done to determine the surface free energy which confirmed that the polished sample had a greater water contact angle as compared to the rough sample, signifying lower surface energy indicating poor wettability, which may result in poor adhesion between the coating and the substrate.

Wear test results showed that, the PEEK coatings deposited on both the rough and polished surfaces did not peel off at the loads of 30 and 40 N respectively even after 10,000 cycles. However, as the load was increased to 50 N, the coating deposited on the polished surface peeled off after approximately 200 cycles of the wear test. This could be attributed to the fact that fewer anchoring sites are available to keep the coating adhered firmly to the substrate because of the polished sample's decreased surface roughness, negative skewness (Rsk) and the lower Rz values of the polished sample along with poor wetting properties. The rough samples, however, did not fail at 50 N until 10,000 cycles (Fig. 1). This can be attributed to the positive skewness of the peak/valley distribution along with good wettability of the surface and increased roughness parameters (Rz, Rq, etc.) which have contributed to the increased adhesion between the coating and substrate. Hence it can be concluded that the rough sample had an improved adhesion as compared to the polished sample, signifying the importance of the surface roughness parameters in determining the effectiveness of a coating.

Table 1. Surface Parameters of the samples.

Parameter (µm)	Polished	Rough
Avg. Roughness (R_a)	0.06094	2.214
Peak Height (R_p)	2.0727	22.222
RMA Avg. (R_q)	0.14	2.7265
Depth of Valley (R_v)	-2.110	-12.656
Max. Height of Profile (R_z)	4.182	34.877
Skewness (Rsk)	-0.1627	0.2135
Kurtosis (Rku)	7.671	2.8022

A study of Ti-doped DLC coating applicable to gears for chassis parts to reduce friction and improve durability

H.S. SHIN [1], Y.K. KIM [1]
[1] Chassis Materials Development Team, Hyundai Motor Group, Hwaseong-si, Korea

Abstract:

DLC coating is mainly composed of carbon and has an intermediate phase between graphite and diamond. It is a chemically stable phase with the slip characteristics of graphite and the high hardness properties of diamond. Due to its outstanding properties, DLC coating is being widely applied and studied in many fields.

Because of the high hardness and intrinsic compressive stress, DLC film is easily spall from the substrates without transition layer (buffer layer, interlayer) or element doping. To improve the properties such as heat resistance, electrical conductivity, and wear resistance, DLC coating is modified by various metal element doping.

In this study, we focused on the mechanical and tribological properties of Ti-doped DLC coating applicable to gears for chassis parts to reduce friction and improve durability.

The coating was deposited using the sputter process. A total of three films were produced to verify the effect of the content of the dopped metal element (based on Ti-C binary phase diagram, carbon content of outermost surface of coating layer is 15, 45, and 75 at.%, respectively). In order to relieve internal stress of the coating layer and to improve adhesion between the interface with the base material, the flow of a carbon source gas was adjusted during the deposition process to form the sloped structure of carbon in the coating layer (Figure 1).

To convince the possibility of Ti-doped DLC coating, we investigated the friction and wear performance compared with two different coatings (Si-DLC, and ta-C). Si-DLC is the material which is widely utilized in various field, and ta-C (tetrahedral amorphous carbon) is specially developed targeting on the its extremely high hardness and excellent low friction characteristics.

The results showed that Ti-doped DLC with a carbon content of 75 at.% in the outermost surface layer reveal stable and comparable friction performance compared to two different coatings.

In particular, in the friction torque evaluation using the FZG gears, Ti-doped DLC with 75 at.% carbon minimizes the wear of counter gear and showed a much superior torque reduction rate across all tested sections.

Keywords: DLC, diamond-like carbon coating, Ti doping, Tribological properties, Gears.

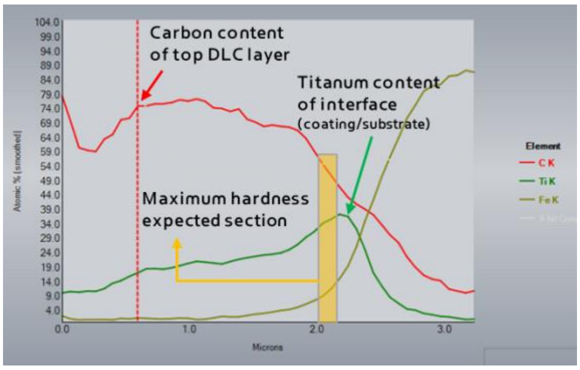


Figure 1: Energy Dispersive x-ray Spectroscopy (EDX) result illustrating content of elements along to depth of Ti-doped DLC: Carbon content varies with depth, forming a sloped structure. Through the maximum hardness expected section in the middle of the sloped structure, adhesion is improved at the interface with the base material.

References:

1. K. Bobzin et al. (2015) Influence of wetting and thermophysical properties of diamond-like carbon coatings on the frictional behavior in automobile gearboxes under elasto-hydrodynamic lubrication, *Surface & Coatings Technology*, **284**, 290-301.
2. L. Qiang et al. (2013) Improving the internal stress and wear resistance of DLC film by low content Ti doping, *Solid State Sciences*, **20**, 17-22
3. J. Cui et al. (2012) Mechanical and tribological properties of Ti-DLC films with different Ti content by magnetron sputtering technique, *Applied Surface Science*, **258**, 5025-5030
4. G. Ma et al. (2012) A study of structure and properties of Ti-doped DLC film by reactive magnetron sputtering with ion implantation, *Applied Surface Science*, **258**, 3045-3050

Experimental study and modeling of tribo-chemical processes in transfer layer during friction in W-C:H coatings in various environments

F. Lofaj [1,*], R. Bureš [1], M. Kabátová [1], H. Tanaka [2,3], Y. Sawae [2,3]
[1] Institute of Materials Research of SAS, Košice, Slovakia
[2] Department of Mechanical Engineering, Faculty of Engineering, Kyushu University, Japan.
[3] Hydrogen Materials Compatibility Division, International Institute of Carbon-Neutral Energy Research, Kyushu University, Japan

Abstract:

Mechanical and tribological properties of nanocomposite W-doped hydrogenated carbon (W-C:H) coatings are controlled by the amount of carbon in the matrix, level of its hydrogenation and environment [1-6]. Depending on the level of hydrogenation of carbon matrix, the coefficients of friction (COF) pure carbon coatings may vary from ~0.5 to superlubricity in dry environments whereas the lowest COF-s in humid air are only around 0.1 [7-8]. Significant COF reduction was attributed to the termination of dangling carbon bonds at the surface by hydrogen [8]. In W-containing carbon coatings, the formation of transfer layer attached to the ball was found to occur [2-3, 9-12]. Thus, COF would be controlled by the formation, composition and shear stresses between transfer layer and coating. In humid air, oxidation of WC, Fe, water vapor dissociation and carbon hydrogenation reactions driven by flash temperatures were identified to occur. In dry or inert atmospheres, some of these reactions would not be possible. Subsequently, the composition of transfer layer would cause different friction behavior. The aim of the current work was to review our earlier results on the formation of transfer layers in HiPIMS W-C(:H) coatings in humid and dry inert environments. These observations were corroborated by modeling based on the minimization of Gibb's free energy energy in the systems with parallel and competing chemical reactions. Modeling was performed in humid air, dry nitrogen, hydrogen and vacuum. The predictions based on modeling were compared with the compositions measured by Raman spectroscopy in the transfer layers generated in the friction tests in the corresponding environments. The agreement between modeling and experimental observations were used to explain the differences in friction behavior in different environments. It was suggested that water vapor in the air controls the level of hydrogenation of carbon originating from WC oxidation. Thus, the amount carbon and its hydrogenation present in transfer layer affects COF. In dry nitrogen, decomposition of WC producing carbon was the dominant reaction. In hydrogen, only the formation of methane from carbon and hydrogen atmosphere was predicted. Thus, assumed carbon hydrogenation would be the dominant reaction. In low vacuum (< 100 Pa), the most important was the decomposition of WC and $FeOWO_3$ into metallic W and Fe.Thus, the variations in the dominant reactions in different atmospheres and subsequently, the amount of lubricating hydrogenated carbon phase in transfer layer could explain the effects of environment on friction behavior in the studied W-C:H coatings.

Keywords: W-C:H coating, coefficient of friction, transfer layer, tribo-chemistry, humid air, dry inert atmosphere.

References:

1. Sánchez-López, J.C., Martínez-Martínez, D., Abad M.D., et al. (2009), Metal carbide/amorphous C-based nanocomposite coatings for tribological applications. *Suf. Coat. Technol.*, 204, 947-954.
2. Makowka, M., Pawlak, W., Konarski, et al. (2016), Hydrogen content influence on tribological properties of nc-WC/a-C:H coatings. *Diamond Relat. Mat.*, 67, 16-25.
3. Nouvellon, C., Belchi, R., Libralesso, L., et al., (2017),WC/C:H films synthesized by an hybrid reactive magnetron sputtering/Plasma Enhanced Chemical Vapor Deposition process: An alternative to Cr(VI) based chromium plating. *Thin Solid Films,* 630, 79-85.
4. Lofaj, F., Kabátová, M., Klich, M., et al. (2019), The comparison of structure and properties in DC magnetron sputtered and HiPIMS W-C:H coatings with different hydrogen content, *Ceram. Int.*, 45, 9502-14.

5. Lofaj, F., Kabátová, M., Klich, M., et al. (2019), Tribological behavior of hydrogenated W-C/a-C:H coatings deposited by three different sputtering techniques, *Cerâmica*, 65, 58-69.
6. Lofaj, F., Kabátová, M., Kvetková, L., Dobrovodský, J., (2020), The effects of deposition conditions on hydrogenation, hardness and elastic modulus of W-C:H coatings, *J. Eur. Ceram. Soc*., 40, 2721-2730.
7. Donnet, C., Grill, A. (1997,) Friction control of diamond-like carbon coatings. *Surf. Coat. Technol*., 94-95, 456-462.
8. Erdemir, A., Eryilmaz, O.L., Nilufer, I.B., et al., (2000), Effect of source gas chemistry on tribological performance of diamond-like carbon films, *Diamond Relat. Mat*., 9, 632-637.
9. Bhowmick, S., Lou, M., Khan, M.Z.U., Banerji, A, Alpas, A.T., (2017), Role of an oxygen atmosphere in high temrperature sliding behavior of W containing diamond like carbon (W-DLC), Surf. Coat. Technol., 332, 399-407.
10. F. Lofaj, M. Kabátová, R. Bureš, (2021), Transfer layer evolution during friction in HIPIMS W–C coatings, Wear, 486-487, 204123.
11. F. Lofaj, H. Tanaka, R Bureš, M. Kabátová, M.; Sawae, Y. (2022), Tribochemistry of Transfer layer evolution during friction in HiPIMS W-C and W-C:H coatings in humid oxidizing and dry inert atmospheres, *Coatings,* 12, 493.F. Lofaj, R. Bureš, M. Kabátová, H. Tanaka, Y. Sawae, (2022), Modelling of tribo-chemical reactions in HiPIMS W-C:H coatings during friction in different environments, *Surf. Coat. Technol*., 434 128238.

Residual stress development and thermo-elasto-plastic deformation in brake discs

Youngjae Kim[1,2] , Myoung-gyu Lee[2, *]
[1] Materials Research & Engineering Center, Hyundai Motor Company, Seoul, Republic of Korea
[2] Department of Materials Science and Engineering & RIAM, Seoul National University, Seoul, Republic of Korea

Abstract:

In this study, the relationship between thermoelastoplastic deformation during braking and residual stress in brake discs was experimentally investigated. X-ray diffraction is performed to verify the residual stress distribution in the depth direction, while the cutting/cutting method is used to quantitatively measure the residual stress over a larger disc area. Thermoelastic strain is also quantified using thermal strain measured using a dynamometer. Compressive residual stresses are distributed on the circumferential and radial surfaces of an unused brake disc with a magnitude of approximately 30 MPa, which after continuous braking tests are converted into tensile stresses exceeding 50 MPa in the circumferential direction, resulting in thermoelastic deformation. . This distortion is proportional to the tensile stress occurring along the braking direction, which is mainly caused by external forces and plastic deformation due to temperature gradients in the disc surface and core. Distortion magnitudes range from 60 to 140 μm depending on the pad material type. Abrasive wear using friction pads can reduce thermoelastic disk distortion by releasing tensile stresses on the plastically deformed disk surface. However, the magnitude of the initial compressive stress has a minor effect on the deformation. This experimental study provides insight into the factors that affect the thermoelastoplastic deformation of brake discs and how the deformation of brake discs can be reduced at the design stage.

Keywords: : Brake disc, thermo-elasto-plastic distortion, residual stress, abrasive wear

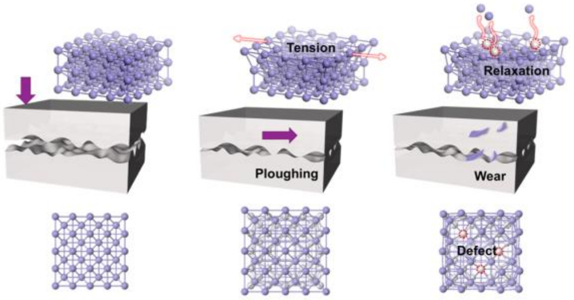


Figure 1: Schematic diagram of abrasive wear by ploughing effect with relaxation of plastic deformation: (a) Initial state (non-braking), (b) local tensile stress caused by plastic deformation during braking, and (c) abrasive wear that breaks interatomic bonds by the ploughing effect, and the resultant relief of tensile stress.

References:

1. Tavangar R, Moghadam HA, Khavandi A, Banaeifar S.(2020) Comparison of dry sliding behavior and wear mechanism of low metallic and copper-free brake pads. *Tribol Int 2020;151:106416.*
2. Weitao S, Bin W, Xiaoliang L, Yuqian W, Jian Z. (2022), Controlling the tribology performance of gray cast iron by tailoring the microstructure. *Tribol Int 2022;167:107343.*

Influence of Coating on the Wear Performance of Wood Cutting Tools

Vanella Mbakop Nanshie[1], , Ali Aidibe[1], Majid Heidari[2] Mohammad Jahazi[1*]
[1] Mechanical Engineering Department, École de technologie supérieure (ÉTS), Montreal, Quebec, Canada
[2] DK-Spec, Quebec, Canada

Abstract:
The forestry industry plays an important role in Canada's regional economy. It employs more than 205,000 people and has been the source of more than $33 billion in exports. In this context, primary and secondary wood processing are of considerable importance, both economically and environmentally. Wood processing involves numerous cutting stages, using tools of different shapes. In order to achieve precise cuts and minimize wood loss, it is essential that the tool be hard, resilient, and wear-resistant. Anti-wear coatings have recently been applied to wood-cutting tools, but the results obtained, while promising, are not consistent. In the present work, the influence of coating on wear damage of three different wood cutting tools, commonly used in industry, is examined. A methodology was developed to accurately and non-destructively quantify the wear damage severity.

Abrasion wear tests according to the ASTM G65 standard revealed that thanks to the coating it was possible to reduce the wear by about 50% for one of the alloys while for another one, the coating did not provide significant improvement. Wear mechanism analyses indicated dominant abrasive wear, with larger chips observed for some steels, a sign of repetitive impact wear. The results suggest a long-term superiority of coated steels and highlight the potential benefits of coatings to increase tool durability.

Keywords:. wear resistance, wear mechanisms, wear test, influence of microstructure on wear, PVD coating, quantification of wear, tool edge, wood cutting, non-contact 3D measurement system, scanning and digitization procedure.

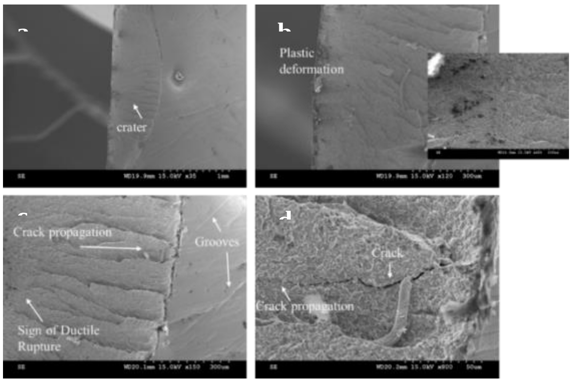


Figure 1: SEM images showing a significant number of cracks near the chipped zones, where cracks are clearly visible around the chipped zones.indicating the lower toughness of this steel compared to the other steels.

Compositionally modulated Ni-W multilayers to alleviate the residual stresses in coatings for superior wear resistance

Nitin P. Wasekar[1*], L. Bathini [1,2], MJNV Prasad [1,2]

[1] Centre for Engineered Coatings, International Advanced Research Centre for Powder Metallurgy and New Materials (ARCI), Hyderabad 500 005, India.
[2] Microstructural Engineering and Mechanical Performance Laboratory, Department of Metallurgical Engineering and Materials Science, Indian Institute of Technology Bombay, Mumbai 400 076, India.

Abstract:

The presence of tensile residual stresses in the Ni-W alloy coatings generated during the electrodeposition has restricted their industrial deployment. In the present study, the multilayering strategy is employed in developing the Ni-W coatings with the best combination of hardness and residual stresses. Ni-W multilayer coatings composed of alternate soft (~ 2.6 GPa) and hard (~ 8.5 GPa) Ni-W layers with individual layer thickness (λ) ranging from 2.5 µm to 0.05 µm were deposited by using the pulse reverse current electrodeposition. These multilayer coatings have demonstrated a significant reduction in the residual stresses while retaining the high hardness. Deconvoluted XRD analysis shows that the soft Ni-W layers have effectively accommodated the residual stresses generated in the hard Ni-W layers. Ni-W multilayer coating with λ ~0.1 µm has exhibited superior resistance to wear damage compared to its homogeneous counterparts. The improved wear degradation resistance of the multilayer coating was rationalized through assessment of residual stresses and thermal properties.

Keywords: Pulse reverse electrodeposition; Ni-W alloy; Multilayer coatings; Microstructure; Wear.

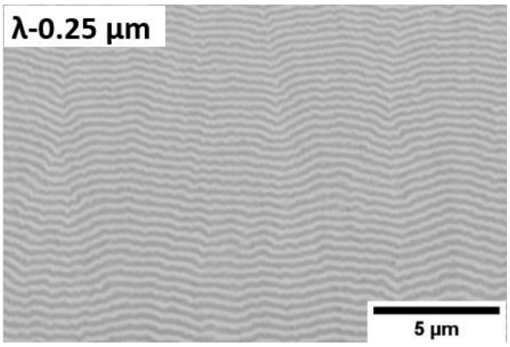
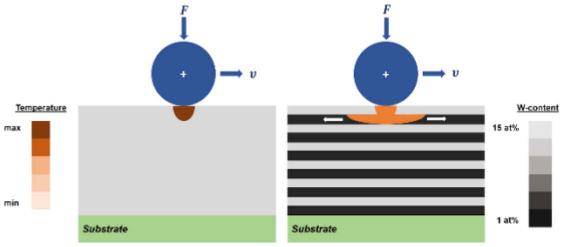


Figure 1: Figure illustrating the Ni-W multilayered coatings with excellent strain hardening ability and reduced residual stress. The multilayered approach led to effective heat transfer through low W layer thereby reducing oxidation and formation of stable oxide film due to friction.



References:

1. Lavakumar B., Prasad M.J.N.V., Phani P.S., Wasekar N.P. (2023) Influence of compositional and microstructural gradient on the wear performance of nanocrystalline Ni–W coatings, Wear, 530-531, 205039
2. Lavakumar B., Prasad M.J.N.V., Wasekar N.P. (2022) Development of continuous compositional gradient Ni-W coatings utilizing electrodeposition for superior wear resistance under sliding contact, Surf Coat Technol, 445, 128728

Tailored Coatings for Enhanced Long-Term Tribological Performance of Journal Bearings in Accelerator-Driven Nuclear Reactors

E.Serag [1,2,*], B. Caers [1], E. Haye [2], P. Schuurmans [1], S. Lucas [2],
[1] Belgian Nuclear Research Centre (SCK CEN), Boeretang 200, B-2400 Mol, Belgium.
[2] Laboratoire d’Analyse par Réactions Nucléaires (LARN), Namur Institute of Structured Matter (NISM), University of Namur, 5000 Namur, Belgium.

Abstract:

This research focuses on improving the tribological performance of journal bearings made of austenitic stainless steel (AISI 316L), intended for use in the MYRRHA accelerator-driven reactor at SCK CEN. The reactor utilizes Lead-Bismuth Eutectic (LBE) as a coolant, which is considered as a bad lubricant due to its low viscosity and poor wettability. The primary objective is to protect the journal bearings against wear during their operational lifespan within the reactor. This is intended to be done through the use of protective layers.

Two distinct coating strategies were explored: TiAlN and carbon-based coatings. The TiAlN coating, known for its high hardness, provides inherent wear resistance. On the other hand, the carbon-based coating offers a low friction coefficient and acts as a solid lubricant through the generation of a transfer layer between the surfaces in contact. Both coatings were applied using advanced techniques – magnetron sputtering deposition for TiAlN and plasma-assisted chemical vapor deposition (PA-CVD) for the carbon-based coating.

This study delves into the deposition processes, characterization of coatings, and their subsequent application in enhancing the wear resistance and lubrication efficiency of austenitic stainless steel journal bearings immersed in LBE. The research aims to contribute insights into effective protective measures for components operating in challenging LBE environments, ultimately advancing the reliability and longevity of journal bearings in such reactors.

Keywords: wear, friction, journal bearings, coatings, LBE, magnetron sputtering, PA-CVD

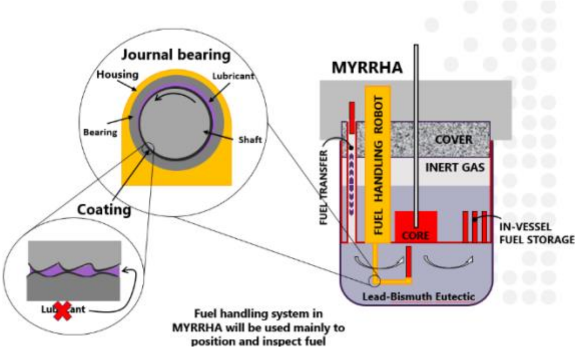


Figure 1: Figure illustrating MYRRHA and the fuel handling system of which the journal bearing is part of. Coating is deposited on the shaft of the bearing to protect it against mechanical wear during its operation inside LBE.

References:

1. E. Serag, B. Caers, P. Schuurmans, S. Lucas, E. Haye, Challenges and coating solutions for wear and corrosion inside Lead Bismuth Eutectic: A review, Surf. Coat. Technol. 441 (2022) 128542.

SICT 2024 Session II. A: Surface and coatings Characterization / Properties Multifunctional composite and hybrid coating

Exploiting the natural instability in thin and flexible dielectric solid films for sensing and photonic applications

S.Freddi[1], M.Gherardi[2], N. Granchi[3], M. Salvalaglio[4], A. Voigt[4], B. Squeo[5], M. Pasini[5], A. Chiappini[1], A. Fedorov[1], M. Abbarchi[6], M. A.Vincenti[7], F. Intonti[3], M. Bollani[1*]
[1] Institute of Photonics and Nanotechnology (IFN) – CNR, LNESS, Como, Italy
[2]Department of Physics, Politecnico di Milano, Milan, Italy
[3]Department of Physics and Astronomy and LENS, University of Florence, Sesto Fiorentino, Italy
[4]Institute of Scientific Computing, Technische Universitat Dresden, Dresden, Germany
[5]Institute of Chemical Sciences and Technologies (SCITEC) – CNR, Milano, Italy
[6]Solnil 95 Rue de la République, Marseille 13002, France
[7]Department of Information Engineering, University of Brescia, Brescia, Italy

Abstract:

Silicon-based nanocrystals represent a promising resource both for next generation electronic devices and for nano-photonics applications. Their exploitation, however, requires precise size, shape and position control [1,2]. Owing to their large surface-area-to-volume ratio, thin semiconductor solid films are often unstable upon annealing. As such, under the action of surface diffusion, the film breaks eventually forming isolated islands when heated at temperature well-below the melting temperature of the bulk material. This phenomenon, known as solid-state dewetting, is one of the main factors impeding the use of ultra-thin silicon films on insulators (UT-SOI) for the further miniaturization of electronic components. Here, we demonstrate the ultimate control of Si and SiGe-based thin films dewetting for the precise formation of complex nano-architectures, and their exploitation as dielectric nano-antennas and field-effect transistors wire. [3,4]. The dielectric antennas are realized exploiting the natural mechanical instability of thin solid films to form regular patterns of monocrystalline atomically smooth Si and Ge nanostructures that cannot be realized with conventional methods. Solid state dewetting (SSD) indeed is a natural shape instability occurring in thin solid films when heated at high temperature: it transforms a flat layer in isolated islands in a timeframe independent from the sample size. However, its potential for applications based on complex pattern formation is still unexplored in spite of the manifold advantages it offers: a) it forms monocrystalline and facetted structures, free from defects and from the typical roughness produced by conventional etching methods; b) the islands are directly formed on an insulating substrate; c) spontaneous dewetting can produce over arbitrary scales patterns that cannot be designed numerically. Therefore, SSD can be efficiently exploited in all these fields to form perfectly ordered and complex nano-architectures over large scales, as well as randomly organized, isolated islands. By a properly combining e-beam lithographic and reactive ion etching processes, we can realize dewetted nanostructures that can play as Mie resonators. As a building material for such resonators Si or SiGe particles are very promising, since their absorption losses are very weak at visible and near-infrared frequency. Furthermore, differently from metallic particles used in plasmonic (e.g., gold, silver, aluminum), they are compatible with silicon-based nanofabrication technologies being, therefore, more appealing for low-cost production and integration in electronic devices.

Keywords: solid state dewetting, SiGe, Mie resonator, flexible photonic.

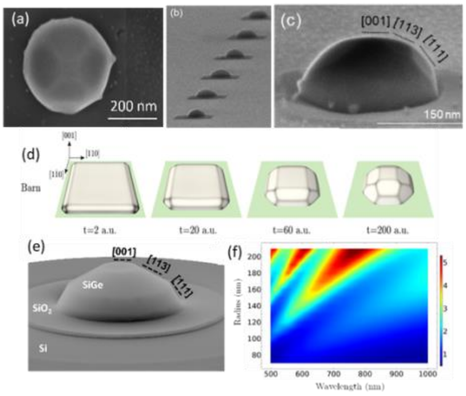


Figure 1: (a) SEM top view and (b) tilted view images of a single dewetted SiGe island. (c) SEM image of a single island where the faceting planes are labelled. (d) simulated evolution of island formation and (f) FEM scattering cross section of the simulated island (e).

References:

1. M. Aouassa et al, Appl. Phys. Lett. 101, 013117 (2012)
2. N. Granchi, et al., APL Photonics 6.12 (2021): 126102
3. M. Naffouti et al, Science Advance, Vol. 3, n°11, eaao1472 (2017)
4. M.Bollani, et al., Nature Comm.,10, 5632, (2019).

Role of Critical Coupling in planar optical nanodevices

E. Popov[1],
[1] Aix Marseille University, Institut Fresnel, 13013 Marseille, France

Abstract:

In 2019, after several years of theoretical, numerical and experimental studies, we obtained the experimental demonstration of second harmonic generation (SHG) in resonant dielectric gratings put inside a Fabry-Perot cavity (CRIGF for Cavity resonator integrated grating filter) based on Lithium Niobate in thin layers. The external resonator gave the possibility to work in focused incident beams without decreasing the resonant strength.

In order to increase significantly the quality factor and SHG signal of CRIGF devices, we propose two methods: The first method uses a geometric asymmetry of CRIGF allowing exciting a quasi-anti-symmetric mode (quasi-dark-mode). The second choice introduces a nano-structuring of the central grating of the CRIGF with a base motif of the bi-atom type (with two lamellae of different widths). The theoretical and numerical studies demonstrated the possibility of increasing the quality factor from several hundreds to more than a million, and the corresponding SHG signal by a factor of 103 to 105. In addition, we demonstrated the existence of two types of operation of the CRIGF: first with the undesirable losses lower than the strength of the coupling/decoupling (over-coupling regime). In this regime, the CRIGF is characterized by a reflection at resonance greater than 90%, which allows it to be used in optical filtering. In the opposite (under-coupling regime), the quality factor increases, but the maximum of the reflection decreases to less than 10%. The transient regime, called critical coupling, is characterized by a reflection of the order of 30-50% and a strong maximum of the electric field inside the resonator.

Keywords: Nanomaterials, Second-harmonic generation, local field enhancement, diffraction gratings, micropatterns.

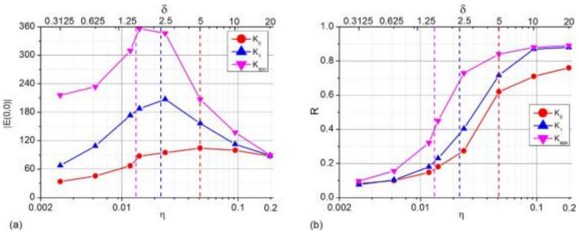


Figure 1: Dependence of (a) the amplitude of the electric field in the middle of the structure, and of (b) the reflectivity maximum as a function of the perturbation parameter, corresponding to the nanostructuring of the bi-atom motive.

References:

1. Popov, E., Hemsley, E., Fehrembach, A.-L., Gothier-Lafaye, O., Monmayrant, A., Calvez, S. (2022) Extreme enhancement of the quality (Q)-factor and mode field intensity in cavity-resonator gratings, *Opt. Express*, 30, 25390-25399.
2. Hemsley, E., Gothier-Lafaye, O., Monmayrant, A., Dubreuil, P., Calvez, S., Fehrembach, A.-L., Popov, E. (2011), Critical coupling in cavity-resonator integrating-grating filters (CRIGFs), *IOpt. Express*, 31, 27274-27286.

Optimization of Surface Reactivity of Diamond Particles by Atomic Layer Deposition of Alumina Coating

K. Shibuta [1*], H. Ishida [1], M. Groner [2], J. Gauspohl [2]
[1] SEKISUI CHEMICAL CO., LTD., Mishima, Osaka, Japan
[2] FORGE NANO, Inc., Thornton, Colorado, USA

Abstract:

Due to the compact size, high power, and high frequencies employed by modern electronic devices, they generate a very large amount of excess heat. To mitigate this problem, thermal interface materials (TIMs) are crucial for efficient heat dissipation to allow proper functioning of devices and extend their lifetime. TIMs are composed of a polymer matrix and thermally conductive filler particles (**Figure 1**). The high thermal conductivity of the filler particles results in efficient heat dissipation.[1, 2)]

Diamond is one of the most promising fillers for TIMs, because it has the highest thermal conductivity of all bulk materials (2200 W/mK).[3)] However, the chemical inertness of the diamond surface is a barrier to combining diamond particles with a polymer matrix, because it suppresses bonding between the particles and the surfactants contained in the matrix. Therefore, there is a strong demand for a methodology for improving the chemical reactivity of the diamond surface without reducing the thermal conductivity of the diamond particles themselves.[4)]

In this study, we attempted to deposit an ultrathin conformal alumina film on diamond particles by atomic layer deposition (ALD) (**Figure 2**). The coated diamond surface was found to exhibit drastically enhanced chemical reactivity. In addition, a TIM fabricated using coated diamond particles and a polydimethylsiloxane matrix exhibited significantly improved thermal conductivity and flexibility. This indicates that the alumina film efficiently reacts with surfactants in the polymer matrix, thus improving the affinity between them. The results of a detailed analysis on alumina film and a reactivity of pristine diamond on ALD will be reported.

Keywords: heat dissipation, thermal conductivity, thermal interface material, diamond particle, polymer matrix, surfactant, atomic layer deposition, alumina, dry coating, surface reactivity

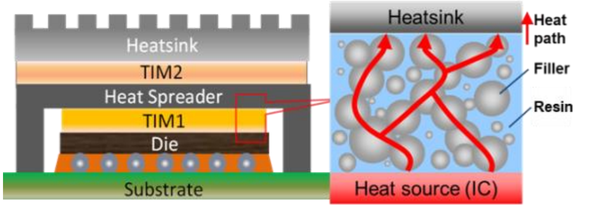


Figure 1: Schematic illustration of heat dissipation mechanism in TIMs. The type of filler has a significant effect on TIM performance because it acts as the main heat-transfer pathway.

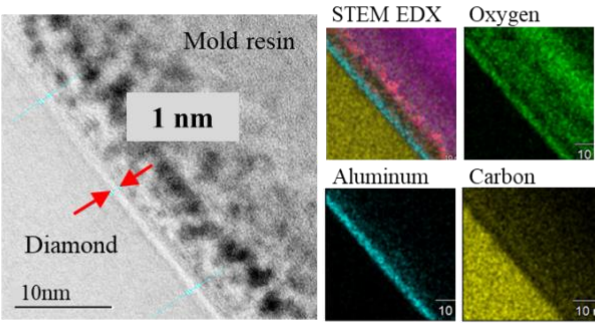


Figure 2: Cross-sectional STEM image and EDX maps showing homogeneous alumina layer on diamond surface.

References:

1. Chen, H., Ginzburg, V.V., Yang, J., Yang, Y., Liu, W., Huang, Y., Du, L., Chen, B. (2016) Thermal conductivity of polymer-based composites: Fundamentals and applications, *Prog. Polym. Sci*., 59, 41-85.
2. Zhang, Y., Ma, J., Wei, N., Yang, J., Pei, Q-X. (2021) Recent progress in the development of thermal interface materials: a review, *Phys. Chem. Chem. Phys.*, 23, 753-776.
3. Prikhodko, D., Tarelkin, S., Bormashov, V. (2016) Thermal conductivity of synthetic boron-doped single-crystal HPHT diamond from 20 to 400 K, *MRS Communications*, 6, 71-76.
4. Ferguson, J.D., Weimer, A.W., George, S.M. (2000) Atomic layer deposition of ultrathin and conformal Al_2O_3 films on BN particles, *Thin Solid Films*, 371, 95-104.

Effect of Particles Dispersion Degree on Microstructure of Composit Plating for Ni-Magetite

T. Mori[1,*], W. Murata[1], K. Nakamura[1], K. Kitamura[1], Y. Tanaka[2], K Fukui[2], A. Fujita[2], M. Takagi[2]
[1] Department of Chemical Science and Technology, Faculty of Bioscience and Applied Chemistry,
Hosei University, Koganei, Japan
[2] Toyohashi Plating Co.,Ltd., Toyohashi, Japan

Abstract:
Recently, demand for electromagnetic wave shield has been increasing in electronics industry and information society. For fabrication of electromagnetic wave shield, magnetic film plating is one of the most important methods, including not only the conventional magnetic alloy plating but also composit plating. In comsit plating, fine particles, especially for nano particles are tried to used in order to further improve the electromagnetic characteristics. However, fine particles are normally difficult to disperse well in a solution with relatively high ion concentration such as plating liquid. Under these circumstances, it is believed that the particles dispersion state in the plating liquid must strongly affect the microstructute of the composit plating film, however, it is hard to properly evealuate the particles dispersion degree in the plating liquid. Therefore the relationship between the particles dispersion degree in the plating liquid and the microstructure of the composit plating film is still unclear.

Thus, the objective of this study is to clarify the effect of dispersant type on the particles dispersion state in the plating liquid and the microstructure of the composite plating film.

Firstry we prepared Ni plating liquid in which magnetite particles were dispersed by using several kinds of dipersants. The nominal size of magnetite particle was 0.2 µm and the particle volumetric concentration was 4.0 vol%. The dispersion degree of magnetite particles in the plating liquid was evaluated by sedimentation test and flow cueve measurement. The zeta potential of magnetite particle before and after adding the dispersant was measured. The Cu plate was immersed in the plating liquid and then applied DC electric field with a current density of 1 or 5 A/dm^2. The composit plating film was analysed by a X-ray fluorescence spectrometer to determin the thickeness of the plating film and the magnetite content in the plating film. The microstructure of the composit plating film was observed by SEM and anlysed by EDX.

Table 1 shows electrophoretic mobility of the magnetite particle before and after addition of dispersants. It was shown that the magnetite particle originally had a positive charge in HCl solution with pH 4, which corresponds to pH of the composit plating liquid but its charge became almost nutral or a little negative in the real composit plating liquid. Furthermore, regardless of dispersant type such as cation, nonion, and anion, the magnetite particle still had a small negative charge when adding the dispersant in the composite plating liquid.

Table 1: Electrophoretic mobility of magnetite particle with and without dispersant.

Dispersant type	Mobility (µm•cm /(V•s))
non (without dispersant)	-0.0711
Dispersant A (cation type)	-0.0612
Sodium 1-Dodecanesulfonate	-0.1017
Polyethylene glycol	-0.1379

Table 2: Effect of dispersant type on magnetite particle content in composites. (5 A/dm^2)

Dispersant type	Magnetite content (mass%)
non (without dispersant)	5.03
Dispersant A (cation type)	6.83
Sodium 1-Dodecanesulfonate	4.82
Polyethylene glycol	6.41

Table 2 shows the effect of dispersant type on the magnetite particle content in the composite plating film. These results indicated that the charge of magnetite particle in the composit plating liquid did not play an important role to control the magnetite content. From the sedimentation test, it was suggested that the particle dispersion degree should affect the magnetite particle content.

Keywords: composite plating, particles dispersion state, zeta potential, electrophoretic mobility, dispersant type, particles content in composite, composite microstructure.

Effect of thermal treatment with different temperature-duration schedules on mechanical and corrosion properties of Ni-P nanocomposite coatings reinforced with ZrO_2 particles

G. Pedrizzetti [1*], E. Baroni [2], M. Merlin [2], G. Pulci [1], F. Marra[1]
[1] Department of Chemical Engineering, Materials, Environment, Sapienza University of Rome, INSTM Reference Laboratory for Engineering of Surface Treatments, Rome, Italy
[2] Department of Engineering (DE), University of Ferrara, Ferrara, Italy

Abstract:

Electroless Ni-P coatings with high P content (≈ 11 wt%) are often employed in the energy production industry to protect steel components from extreme environmental attack, including corrosion and wear phenomena. Properties and durability of these coatings and can be improved by two strategies: (i) heat treatment; (ii) introduction of functional second-phase particles to create a composite coating. When the first strategy is adopted, growth of Ni grains and precipitation of Ni_3P phases effectively increase coating hardness. Nevertheless, Ni crystallization might negatively affect corrosion resistance [1] and decrease the fracture toughness of coatings [2]. To better understand properties evolution according to microstructural changes, this work focuses on the manufacturing of HP coatings reinforced with ZrO_2 nanoparticles and characterization after heat treatment at different temperature-time schedules, to define the best conditions in which reinforcing nanoparticles and annealing can act synergistically to maximize performance.

Two temperatures of heat treatment were selected, 340°C and 400°C, and duration was varied between 0.5 h and 8 h. Microstructural evolution after nanoparticles introduction and annealing was evaluated by XRD analysis. Evolution of properties was characterized in terms of Vickers microhardness, elastic modulus and fracture toughness (measured by instrumented indentation), and corrosion resistance (investigated by potentiodynamic polarization test).

Results demonstrated that the amorphous microstructure of HP coatings can be maintained after heat treatment at 340°C up to 2 h duration. Conversely, 0.5 h is sufficient for crystallization when annealing is performed at 400°C. Further exposure at 400°C leads to the growth of both Ni grains and Ni_3P precipitates, whereas treatment at 340°C promotes precipitation while keeping grain size relatively limited. Comparable results were obtained for both standard and nanocomposite coatings, confirming that nanoparticle introduction does not alter the evolution of microstructure. The best results of coatings microhardness were obtained for nanocomposites annealed at 400°C for 1h and 340°C for 8h, with ≈80% microhardness increase compared with standard HP. This demonstrates an effective synergistic effect of dispersion hardening (given by nano-ZrO_2), precipitation strengthening (given by Ni_3P) and grain growth (according to the i*nverse* Hall-Patch equation) [3]. Both the introduction of nanoparticles and thermal treatments lead to an increase of the elastic modulus of coatings, with maximum value obtained after exposure at 400°C for 1 h. As expected, fracture toughness dramatically decreases after heat treatment. Slightly higher values of K_I can be obtained after treatment at 380°C for 8 h compared to 400°C for 1 h. This different behavior can be ascribed to the different microstructure of the former, with the combined effect of finer grain size and, especially, coarser precipitates [2]. Further increase in K_I can be registered as a result of nanoparticle addition. Corrosion resistance of coatings can be significantly improved by the introduction of ZrO_2 inert nanoparticles, due to the reduction of surface area active for corrosion. Further improvements are given by heat treatments since precipitation of Ni_3P phases enhances the passive behavior of coatings.

To conclude, the synergistic effect of nano-ZrO_2 introduction and microstructural changes by heat treatment can be an effective strategy to enhance properties of NiP coatings, making them suitable for advanced engineering applications.

Keywords: electroless deposition, nanocomposite Ni-P coatings, corrosion resistance, microhardness, elastic modulus, fracture toughness, microstructure, harsh environment applications

References:

1. Shu X. et al. (2015) *Surf Coat Technol*, 261, 161–166.
2. Sade W. et al (2011), *ISRN Materials Science*, 1-6.
3. Pedrizzetti G. et al. (2023), *Chem Eng Trans*, 100.

Templating effects of graphene in stabilizing ultrathin compounds on metals

A. Brambilla[1,*], A. Picone[1], M. Capra[1], S. Fiori[2], D. Dagur[2], G. Vinai[2], G. Panaccione[2], and F. Ciccacci(1)
[1] Dipartimento di Fisica, Politecnico di Milano, Piazza Leonardo da Vinci 32, Milano, Italy
[2] Istituto Officina dei Materiali (IOM)-CNR, Laboratorio TASC, Trieste, Italy

Abstract:

Ultrathin oxide films grown on metallic substrates have been intensively investigated in modern surface science [1,2]. A convenient way to tailor the physical and chemical properties of oxide/metal interfaces is the use of a buffer layer, i.e. an ultrathin metallic or oxide film interposed between the substrate and the overlayer. In some of our recent investigations, we have shown that the morphology and electronic properties of oxide films can be tuned by using a buffer layer of graphene stabilized on the Ni(111) surface [3]. High quality graphene layers can, in fact, be successfully grown on the close-packed surfaces of many transition metals [4].

A common strategy to perform a well-controlled growth of thin transition metal oxides, is the reactive deposition of transition metals in an oxygen atmosphere. We have investigated, in this respect, the templating effects of graphene/metal surfaces in stabilizing such low-dimensional compounds. In particular, we have been growing Chromium and Fe oxides on the graphene-covered and graphene-free Ni(111) surfaces and Chromium oxide on the graphene-covered and graphene-free Pt(111) surface.

Considering first the Ni(111) substrate, dramatically different results are obtained for the growth of Cr and Fe oxides. In the former case, an ordered Cr_2O_3 ultrathin overlayer can be obtained, showing that graphene can act as an ideal buffer layer for the growth of high-quality oxide layers [3]. In the latter case, the presence of graphene induces the switching from a 2D to a 3D growth of FeO_x [5], suggesting an effective strategy to maximize the possible catalytic effects of an ultrathin oxide.

Coming to Pt(111), here a multidomain graphene layer could be obtained. Despite this, we were able to show that Cr_2O_3 ultrathin films grown on graphene/Pt(111) maintain their stoichiometric and electronic properties mostly unaltered, compared to Cr_2O_3 on bare Pt(111). On the other hand, a 3D growth scenario is observed for Cr_2O_3 on graphene/Pt(111), and a 2D layer-by-layer growth is observed on bare Pt(111), oppositely with respect to the Cr_2O_3/Ni(111) case and more similarly to the FeO_x/Ni(111) case [6].

I will finally be discussing potential applications of these ultrathin oxides for spintronics applications, making reference also to other classes of hybrid interfaces involving transition metal oxides and molecular materials [7].

Keywords: transition metal oxides,.graphene, surfaces and interfaces, ultrathin films, metal-oxide interfaces.

1. Netzer, F. P.; Surnev, S. Oxide Materials at the Two-Dimensional Limit; Springer, (2016).
2. Picone, A.; Riva, M.; Brambilla, A.; Calloni, A.; Bussetti, G.; Finazzi, M.; Ciccacci, F.; Duò, L. (2016) Reactive metal–oxide interfaces: A microscopic view. *Surf. Sci. Rep.* 71, 32–76.
3. Lodesani, A. ; Picone, A.; Brambilla, A.; Giannotti, D.; Jagadeesh, M.S.; Calloni, A.; Bussetti, G.; Berti, G.; Zani, M.; Finazzi, M.; Duò, L.; Ciccacci, F. (2019) Graphene as an Ideal Buffer Layer for the Growth of High-Quality Ultrathin Cr_2O_3 Layers on Ni(111). *ACS Nano* 13, 4361-4367.
4. E. Voloshina and Y. Dedkov (2012) *Phys. Chem. Chem. Phys.* 14, 13502.
5. Lodesani, A.; Picone, A.; Brambilla, A.; Finazzi, M.; Duò, L.; Ciccacci F.; 3-dimensional nucleation of Fe oxide induced by a graphene buffer layer (2020) J. Chem. Phys. 152, 054706.
6. Fiori S.; Dagur, D.; Capra, M.; Picone, A.; Brambilla, A.; Torelli, P.; Panaccione, G.; Vinai, G. (2023) Electronically ordered ultrathin Cr_2O_3 on Pt(111) in presence of a multidomain graphene intralayer. *Appl Surf Sci*, 613, 155918.
7. SINFONIA FET project, grant n. 964396 - www.sinfonia-fet.eu

Ultralight and robust current collectors for high-energy and high-safety lithium-ion batteries

Suihua He*, Ping Gao

Advanced Materials Thrust, Function Hub, The Hong Kong University of Science and Technology (Guangzhou), Nansha, 510000, China

Abstract:
Polyolefin-based membranes are extensively utilized in the domain of current collectors for "metal-polymer-metal" tri-layered composite structures due to their advantageous blend of low cost, superior mechanical properties, and chemical stability. However, the inherent nonpolar nature of polyolefin-based thin films, while conferring excellent chemical stability, simultaneously results in suboptimal adhesion to metals. Furthermore, the conventional thermal instability polyolefin-based membrane might shrinkage when subjected to elevated temperatures, which can cause short circuits, thermal runaway, and thus explosions in the battery system. Aramid nanofibers (ANF), as organic material that can be coated independently on the polyolefin-based membrane, has a good consistency, light weight, and good thermal stability[3]. Herein, ANF protective layer reinforced nanoporous ultrahigh molecular weight polyethylene film (UPE) is constructed. In addition, magnetron sputtering is used to deposit an ultra-thin copper layer on the ANF-UPE membrane. The resulting Cu/ANF-UPE/Cu membrane can successfully surpass the thermal shrinkage and puncture of this metallized plastic current collector (MPCC) under different input power of the magnetron sputtering process. This flyweight MPCC (0.32 mg cm^{-2}) achieves exhibits an excellent tensile strength (~300MPa) with a strain of 40% (commercial copper foil broke at a strain of 2.7%), and achieves no thermal shrinkage under 180℃. Overall, an ultralight and thermomechanically robust cellular polymer membranes was developed, which highlights the plausible processing scalability for satisfying the requirements of lithium battery systems with high-level safety and long-term cycling performance under elevated temperatures.

Keywords: aramid nanofiber, nanoporous ultrahigh molecular weight polyethylene, current collectors, lithium-ion batteries

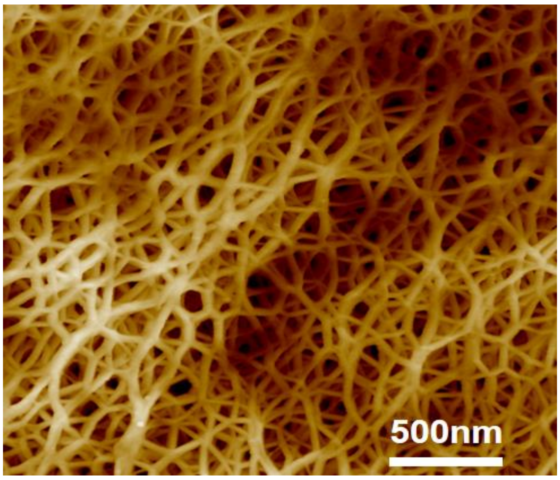


Figure 1: AFM topography of the ultrahigh porosity ANF-UPE membrane.

References:

1. Y. Ye, R. Xu, W. Huang, H. Ai, W. Zhang, J.O. Affeld, A. Cui, F. Liu, X. Gao, Z. Chen, Quadruple the rate capability of high-energy batteries through a porous current collector design, Nature Energy (2024) 1-11.
2. S. Jin, Y. Jiang, H. Ji, Y. Yu, Advanced 3D current collectors for lithium-based batteries, Advanced Materials 30(48) (2018) 1802014.
3. S.Z. Zhang, Y. Song, B. Zhang, L. Wang, X. He, Metallized Plastic Foils: A Promising Solution for High-Energy Lithium-Ion Battery Current Collectors, Advanced Energy Materials 13(36) (2023) 2302134.

Growth behaviour of energy-efficient protective black PEO coatings on additively manufactured AlMgSi alloys

Muhammad Ahsan Iqbal, Miguel Collado Vian, Raul Arrabal, Endzhe Matykina, Marta Mohedano
Departamento de Ingeniería Química y de Materiales, Facultad de Ciencias Químicas, Universidad Complutense de Madrid, 28040 Madrid, Spain

Abstract:

The applicability of energy-efficient flash black PEO coatings on additive-manufactured AlSi10Mg fabricated by SLM (selective laser melting) has been investigated as a solution for protecting aerospace interior components that require corrosion-resistant adhesive black coatings. The effects of different PEO treatment times (1-5 min) were studied in an electrolyte composed of phosphate, Na-EDTA and sodium hydroxide base solution with ammonium metavanadate as colouring additives. The formation of different Vox compounds, which is the key factor that gives the coating its black colour, was influenced by the PEO treatment duration. Specifically, the ratio of black V_2O_3 to brown V_2O_5 was observed to be higher with the increase in PEO treatment time. To better understand the interaction between oxide formation /black colour transition, and the relationship between the PEO composition, structural analysis, and energy consumption of final coatings in association with corrosion resistance properties were also evaluated. The findings showed that with the increase of treatment time from 1 to 4 minutes, the colour of the coatings gradually changes from grey to strong black, due to an increase in the V_2O_3 to V_2O_5 ratio; however, further increases have no or negative effect on the coating's black colour. The corrosion resistance characteristics also became lower at longer PEO treatment time (5 min) due to the intensity of the spark discharge, which is also evidenced by surface roughness analysis, where the high smoothness of the surface is found for 4-min treatment and after-date surface roughness (Sa) increased from 1.95 to 2.11µm^2. Moreover, bare AlMgSi corrosion resistance is enhanced by all-prepared black PEO coatings. The coating with 4-min PEO treatment is the one with the best corrosion characteristics where an impedance modulus of $|Z|_{0.01}$ 1×10^5 Ω.cm^2 was obtained after 7 days of immersion in 3.5% NaCl electrolyte. In addition, the mentioned coating showed energy consumption of 2.05 (kWhm^{-2}µm^{-1}) with distinctive black colour (L-20.9: a-1.3:b-1.9/ CIELAB colour space model). The overall system hardness (Vickers indentation method) increased after black PEO up to 4 orders of magnitude compared with the bare substrate. The developed thin black flash PEO coating was found to have lowered energy consumption while offering substantial corrosion resistance to the underlying substrate, taking into account the film thickness (5-6 ±0.8 µm film thickness) and energy consumption (1.03 to 2.20 (kWhm^{-2}µm^{-1})). The findings of these investigations unequivocally show that the low-energy PEO method herein described is very appropriate for use as a solar absorber coating in space and related applications.

Keywords: Flash PEO, Black coating, Energy consumption, EIS, Aerospace applications.

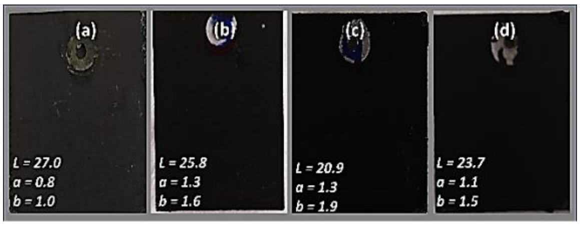


Figure 1: Macroscopic image of PEO-treated specimens: (a) PEO-2 min, (b) PEO-3 min, (c) PEO-4 min, (d) PEO-5 min.

SICT 2024 / Plasma Tech 2024 Joint session II. B: Bio-interfaces, Biomedical / Bioactive surfaces and coatings Plasma applications for biology, medicine, and agriculture

Rapid inactivation of viruses in water by plasma treatments

G. Primc [1,2], M. Zver [1], A. Filipič [3], D. Dobnik [3], M. Mozetič [1,2]
[1] Department of Surface Engineering, Jozef Stefan Institute, Ljubljana, Slovenia
[2] Plasmadis Ltd., Ljubljana, Slovenia
[3] National Institute of Biology, Ljubljana, Slovenia

Abstract:

Alternative methods for the inactivation of viruses in water have recently attracted the scientific community's attention. The methods should enable rapid inactivation at room temperature and should be free from chemicals. The application of gaseous plasma fulfills these regulations but may be impractical because plasma sustained at atmospheric pressure is limited to the volume of the large electric field.

A useful method for sustaining plasma at low pressure despite the water being at atmospheric pressure is the application of hydrodynamic cavitation. The saturated water vapor pressure at the water room temperature is established in a stable supercavitation bubble of volume several cm^3, and the electrodes are immersed into a bubble and powered with a suitable voltage supply to sustain the non-equilibrium gaseous plasma in a glow discharge mode. A preferred embodiment is illustrated in Figure 1. Plasma sustained in water vapor at the pressure of a few 10 mbar is a rich source of OH radicals, which are dissolved in liquid. The high speed of liquid water passing the cavitation nozzle enables optimal mixing, so a few passages of water enable the inactivation of viruses for 5 decades by an irreversible interaction of OH radicals with organic matter. The water pH does not change significantly, and the concentration of hydrogen peroxide remains below 1 mg/L, so the effect of H_2O_2 on virus inactivation is marginal. The preferred discharge power is between 10 and 100 W.

An alternative to hydrodynamic cavitation is the irradiation of water at atmospheric pressure with vacuum ultraviolet (VUV) radiation. An extensive source of VUV radiation is inductively coupled radiofrequency (RF) hydrogen plasma sustained in the H mode. As much as about 10% of discharge power is converted to VUV radiation in the preferred range of discharge parameters. A VUV transparent window separates low-pressure plasma from the liquid water, and the concentration of active viruses drops for 5 orders of magnitude within a minute of treatment. The rapid inactivation in this configuration is also attributed to the irreversible interaction of viruses with OH radicals.

Keywords: plasma, virus, inactivation, water, cavitation, vacuum UV radiation, OH radicals.

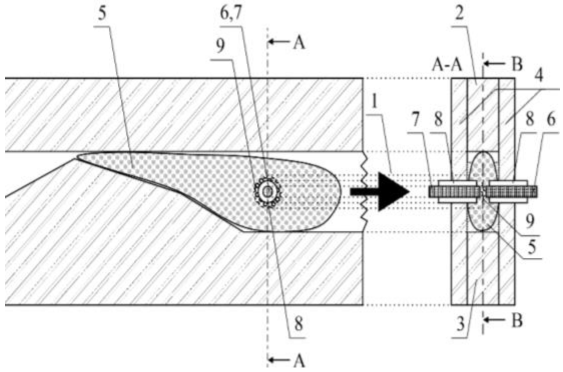


Figure 1: Figure illustrating the formation of a bubble of water vapor (5) inside liquid water at atmospheric or higher pressure. Electrodes (6, 7) are immersed into the bubble and sustain stable plasma in the bubble. The arrow (1) indicates the direction of the water flow.

References:

1. Filipić, A., Gutiérrez-Aguirre, I., Primc, G., Mozetič, M., Dobnik, D. (2020) Cold plasma, a new hope in the field of virus inactivation, *Trends in biotechnology,* 38, 1278-1291.
2. Zver, M., Dobnik, D., Zaplotnik, R., Mozetič, M., Filipić, A., Primc, G. (2023) Non-thermal plasma inactivation of viruses in water solutions, *J. Water Process Eng.* 2023, Vol. 53, Str. 103839-1-103839-12.
3. Primc, G., Zaplotnik, R., Mozetič, M., Filipić, A., Gutiérrez-Aguirre, I., Dobnik, D., Dular, M., Petkovšek, M. (2023) Method and device for disinfection of liquid, *US 11,807,555 B2*.
4. Filipić, A., Dobnik, D., Gutiérrez-Aguirre, I., Ravnikar, M., Košir, T., Baebler, Š., Štern, A., Žegura, B., Petkovšek, M., Dular, M., Mozetič, M., Zaplotnik, R., Primc, G. (2023) Cold plasma within a stable supercavitation bubble - a breakthrough technology for efficient inactivation of viruses in water *Environment international* 182, 108285-1-108285-10.

Well-defined poly(2-isopropenyl-2-oxazoline) brushes provide enhanced biocompatibility and versatility in surface functionalization

Manisha Singh, Lenka Poláková, Andres de los Santos Pereira, Ognen Pop-Georgievski, Jan Svoboda, Tomáš Riedel, Sachin Gupta, Zdeňka Sedláková, Vladimír Raus and Rafał Poręba*
Institute of Macromolecular Chemistry, Czech Academy of Sciences, Heyrovského nám. 2, 162 00 Prague 6, Czech Republic

Abstract:

Poly(2-isopropenyl-2-oxazoline) (PIPOx), a biocompatible polymer[1] amenable to clean and orthogonal post-polymerization modifications, has recently emerged as a suitable candidate for the preparation of functional polymer brushes via surface-initiated reversible-deactivation radical polymerization (SI RDRP). However, the field currently lacks a universal SI RDRP method that would provide a straightforward control over the PIPOx brush thickness and be applicable to non-planar surfaces. Herein, we designed an aqueous, metallic copper-mediated RDRP (Cu(0)-RDRP) protocol for SI grafting of IPOx that manifests an excellent degree of temporal control over the PIPOx brush thickness up to more than 100 nm. The superior kinetic control was achieved through the use of an all-chlorine initiation/catalytic Cu(0)-RDRP system and careful ligand selection, demonstrating a clear advantage over previous approaches based on brominated initiators.[2] Additionally, we found that using neat water as a reaction medium for the Cu(0) catalyst generation in the standard disproportionation step significantly accelerates the brush growth. Importantly, a surface plasmon resonance analysis demonstrated for the first time the high resistance of PIPOx brushes against non-specific protein fouling, as documented by a significant (96 %) decrease in protein deposition from undiluted blood plasma and negligible adsorption from fetal bovine serum and other protein solutions. Finally, we showcased in model scenarios the versatility of the prepared well-defined PIPOx brushes by modifying them with suitable functional carboxylic acids under mild conditions in order to subsequently synthesize graft copolymer brushes or trigger a CuAAC click reaction. Our results highlight PIPOx brushes as an attractive platform for the fabrication of low-fouling, multifunctional surfaces.

Keywords: poly(2-isopropenyl-2-oxazoline), polymer brushes, surface functionalization, protein fouling, low-fouling coating, graft copolymer brush, Cu-RDRP, click reaction

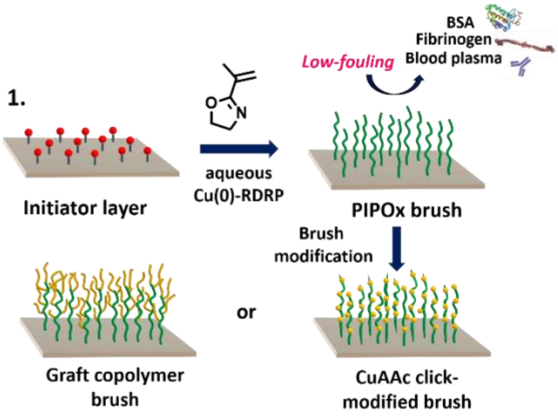


Figure 1: Figure illustrating the modification of Si surface with PIPOx brushes and their subsequent utilization as a platform for straightforward and universal post-polymerization modification exemplified via model CuAAC click reaction and by preparation of graft-copolymer brushes.

References:

1. Z. Kronekova, M. Mikulec, N. Petrencikova, E. Paulovicova, L. Paulovicova, V. Jancinova, R. Nosal, P.S. Reddy, G.D. Shimoga, D. Chorvat, Jr., J. Kronek, Ex Vivo and In Vitro Studies on the Cytotoxicity and Immunomodulative Properties of Poly(2-isopropenyl-2-oxazoline) as a New Type of Biomedical Polymer, Macromol. Biosci., 16 (2016) 1200-1211.
2. V. Raus, A. Hološ, J. Kronek, J. Mosnáček, Well-Defined Linear and Grafted Poly(2-isopropenyl-2-oxazoline)s Prepared via Copper-Mediated Reversible-Deactivation Radical Polymerization Methods, Macromolecules, 53 (2020) 2077-2087.

Microstructure and Properties of Micro-Arc Oxidized Coatings Produced on Plastically Deformed Titanium

A. Trelka-Druzic [1*], F. Muhaffel [2], A. Jarzębska [1], G. Cempura [3], M. Kulczyk [4], H. Cimenoglu [2], Ł. Maj [1]
[1] Institute of Metallurgy and Materials Science, Polish Academy of Sciences, Kraków, Poland
[2] Istanbul Technical University, Istanbul, Turkey
[3]AGH University of Science and Technology, Krakow, Poland
[4]Unipress-Extrusion, Warsaw, Poland

Abstract:
Micro-arc oxidation (MAO) of titanium-based materials is one of many surface modification processes that can provide porous and adhesive, oxide coatings not only increasing their tribological performance but also enhancing bioactivity resulting in more improved osseointegration. Beside that, the MAO is an effective, low-cost and environmentally friendly method making its promising for the use in the area of medical implants [1]. During the MAO process, the specimen and electrolyte pool act as the anode and cathode, respectively. When applying a high voltage between two electrodes, the intense spark discharging takes place on the surface of the specimens (when exceeding the breakdown voltage of forming oxide layer). The latest literature reports showed that plastic deformation of the substrate material strongly accelerates the growth of the coating [2,3]. However, the mechanisms of formation of the coatings on such substrates attracted only a little attention in the past. In this work, titanium oxide-based coatings were deposited with the MAO method on the surface of hydrostatically extruded titanium grade 4. The specimens were MAO-processed for 3 min in an electrolyte composed of an aqueous solution with different amounts of gram per litre of Na2HPO4 and KOH. The positive and negative electric pulses of 150 V-30 V or 175 V-35 V respectively, were applied by pulsed power supply, and the electrolyte temperature was kept below 22 °C. The investigations encompassed microstructural observations, phase and chemical analysis and determination of tribological properties of coatings. The surface morphology and micro-structure of coating cross-sections were analysed using a scanning electron microscope. The thickness of the coatings was measured using ImageJ software based on the coating's cross-section images. Transmission electron microscopy (TEM) was performed to investigate the structural elements at the nanoscale. The TEM samples were cut out with a focused ion beam technique. The analysis of phase composition was performed with an X-ray diffractometer. Using a combination of various parameters for the production of MAO coatings resulted in visible changes in their microstructure features, thickness, surface roughness as well as phase composition. The MAO coating's SEM cross-sectional study confirmed its good adhesion to the substrate, what was presented in Figure 1.

Keywords: micro-arc oxidation, coatings, plastically deformed titanium, microstructure, phase composition, biomedical applications.

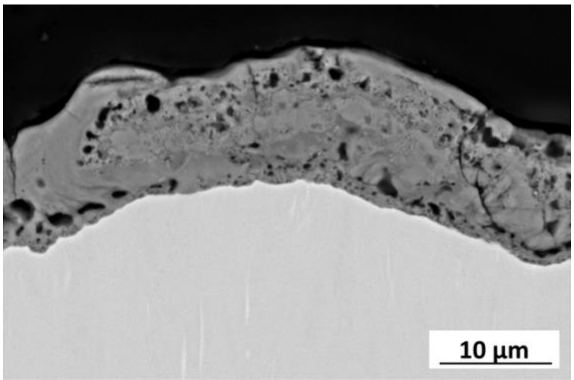


Figure 1: SEM-BSE microstructure of the cross-section of the coatings produced with applied voltages 175 V-35 V and electrolyte containing 12 g/l Na_2HPO_4 and 1 g/l KOH.

Acknowledgment:
This research was financially supported by the The National Centre for Research and Development of Poland, grant number LIDER13/0175/2022.

References:

1. Ł. Maj, et al., Unveiling the mechanisms of coating formation during micro-arc oxidation of titanium in Na_2HPO_4 electrolyte, *Surf. Coat. Technol.*, 473 (2024) 130224.
2. E. Parfenov, et al., Surface functionalization via PEO coating and RGD peptide for nanostructured titanium implants and their in vitro assessment, *Surf. Coat. Technol.* 357 (2019) 669–683.
3. Ł. Maj, et al., Titania coating formation on hydrostatically extruded pure titanium by micro-arc oxidation method, *J. Mater. Science Technol.* 111 (2022) 224-235.

Atmospheric cold plasma technology advancements: enabling cost-effective, standardized and enhanced biomolecule immobilization for biosensing applications

L. Dankers [1,2], K. Leirs [1], B. Nisol [2], J. Lammertyn [1]
[1]Department of Biosystems, Biosensors Group, KU Leuven, Leuven, Belgium
[2]Molecular Plasma Group, Leuven, Belgium

Abstract:

Controlled immobilization of biomolecules is pivotal for the excellent performance of biosensors, which are being used in multiple application fields. In the complex matrices that need to be analyzed, achieving precise capture of target analytes with high specificity and sensitivity is crucial [1]. This study focuses on the plasma-based modification of polymer surfaces typically used for fabricating plastic lab consumables. Due to unique attributes of each surface, identifying a single methodology for the surface functionalization poses a scientific challenge [2]. This emphasizes the need for a standardized immobilization method that allows for improved target detection on a variety of surfaces. Additionally, traditional deposition techniques pose challenges due to the intricacy of manufacturing processes and the potential compromise of biofunctionality [3]. A scalable deposition technology is the cold atmospheric plasma technology developed by the Molecular Plasma Group. It combines the advantages of atmospheric plasma surface activation with the ability to graft molecules to any surface in a single process step. We exploit the Molecular Plasma™ technology to standardize the immobilization of functional and uniform biomolecule layers on a variety of polymer substrates by means of a plasma-deposited carboxylic acid linker-layer. The linker-layer enables standardized immobilization of proteins using EDC/NHS coupling chemistry, with respect to sensitive, specific, reliable, and robust target detection afterward. The functionality of immobilized proteins was compared to the functionality of proteins immobilized using physical adsorption, the gold standard wet-chemistry method. We investigated the stability of the deposited coating, the specificity and sensitivity of the immobilized proteins related to target detection, and, reproducibility of the ELISA-based bioassay. The results indicated that receptors immobilized via a plasma-deposited linker-layer showed increased target detection compared to the traditional immobilizing method (Figure 1A). Additionally, results showed that the plasma-deposited linker-layer generated a stable and reliable surface for reproducible and specific protein immobilization on different materials (PMMA, PVC, glass) (Figure 1B). These results open the perspective towards the application of this novel biofunctionalization approach, which allows biosensors fabrication with a highly performant bioreceptor layer at affordable cost. Hence, this technology enables biosensor commercialization and can revolutionize health care and other fields.

Keywords: cold atmospheric plasma, protein immobilization, polymer surfaces, ELISA-based bioassay, standardization, biomedical applications.

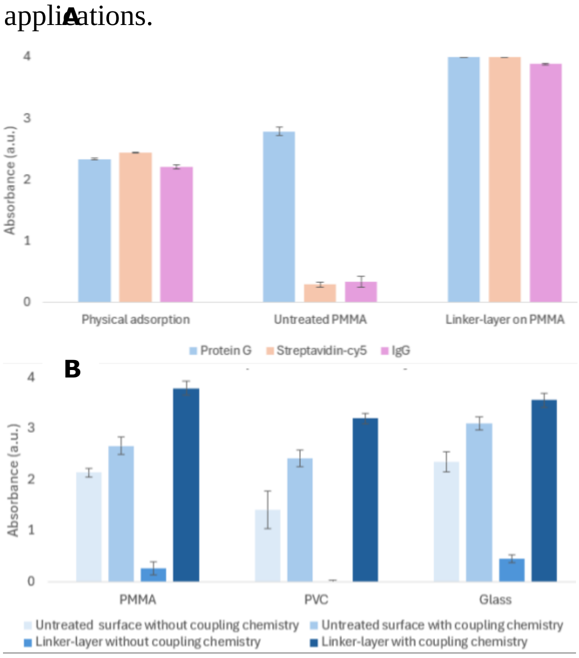


Figure 1: Comparison of physical adsorption in microtiter plate to plasma-based linker-layer on PMMA for protein immobilization (**1A**). Protein G immobilization to multitude of surfaces with- and without EDC/NHS coupling chemistry (**1B**).

References:

1. Naresh, V., Lee, N. (2021) A Review on Biosensors and Recent Development of Nanostructured Materials-Enabled Biosensors, *Sensors.*, 21, 1109.
2. Tsao, C. (2016) Polymer Microfluidics, *Micromachines.*, 7, 225.
3. Panday, CM., Augustine, S., Kumar, S., et al. (2028) Microfluidics Based Point-of-Care Diagnostics, *Biotechnology Journal.*, 13, 1.

Plasma Activated Media for Prostate Cancer Treatment

M. Moreau[1, *], I. Ghzaiel[1], K. Charlet[1], S. Menecier[1], F. Perisse[1], M. Sleiman[2], M. Sarakha[2]
[1] Université Clermont Auvergne, CNRS, Clermont Auvergne INP, Institut Pascal, F-63000, Clermont-Ferrand, France
[2] Université Clermont Auvergne, CNRS, Clermont Auvergne INP, Institut de Chimie de Clermont-Ferrand, F-63000, Clermont-Ferrand, France

Abstract:

Cold Atmospheric Plasma (CAP) is an ionized gas generated at atmospheric pressure with the temperature of particles (molecules, ions, atoms) close to the room temperature. Recent studies have shown that both *in vitro* and *in vivo* plasma exposure to many cancer cell lines is efficient to induce cell deaths through apoptosis pathways [1]. Moreover, non-tumoral cell lines seem to be less impacted by CAP than cancer cell lines: this is called selectivity of plasma [2]. It is highly likely that complex chemical kinetics inside cold plasma coupled with interactions on biological targets lead to formation of Reactive Oxygen and Nitrogen Species (RONS) that cause an important oxidative stress and result in cell deaths through uncleared molecular mechanisms [3]. Direct plasma treatment represents most investigations, but some papers claim that the indirect treatment called Plasma-Activated Medium (PAM) also triggers tumor cell deaths [4]. Therefore, RONS excluding shortest lifetime species remain the only actors for PAM treatment.

In this study, a plasma jet device will be used in controlled conditions to quantify RONS production in the gas phase and into water and/or culture medium (supplemented RPMI-1640).

The experiments will be made for some species in liquid phases irradiated by plasma and in the surrounding gas phase. In liquid phases, a look will be taken for hydroxyl radical ($^{\bullet}OH$), singlet oxygen ($^{1}O_2$), hydrogen peroxide (H_2O_2), nitrites (NO_2^-) and nitrates (NO_3^-). Two liquids will be used as targets: ultra-pure water to have an absolute quantification of species production and culture medium to have a relevant quantification close to *in vitro* experiment conditions. In parallel, ozone (O_3) and nitric oxides (NO_x) production will be measured in gas phase. The importance of some plasma physical parameters on this production will be established. Finally, a link between this chemical study of cold atmospheric plasma and the impact on our future biological assays orientation will be exposed.

Keywords: Cold atmospheric plasma, reactive oxygen and nitrogen species, chemical quantification, liquid phase, gaseous phase.

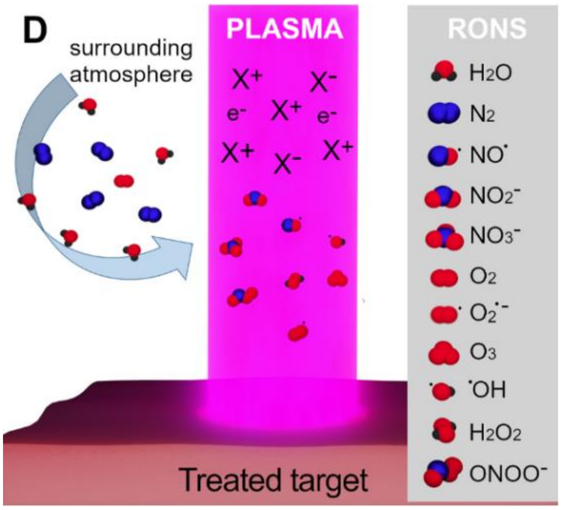


Figure 1: Scheme of reactive oxygen and nitrogen species at plasma-biological target interface. All of them may have a key role on cell deaths through oxidative stress by mechanisms that are still uncleared. Figure from Busco *et al.* [3].

References:

1. Min, T., Xie, X., Ren, K., Sun, T. Wang, H., Dang, C., Zhang, H. (2022) Therapeutic Effects of Cold Atmospheric Plasma on Solid Tumor, *Frontiers in Medicine*, 9.
2. Fofana, M., Buñay, J., Baron, S., Menecier, S., Nivoix, M., Perisse, F., Vacavant, A., Balandraud, X. (2019) Selective treatments of prostate tumor cells with a cold atmospheric plasma jet, *Clinical Plasma Medicine*, 17-18, 100098.
3. Busco, G., Robert, E., Chettouh-Hammas, N., Pouvesle, J.-M., Grillon, C. (2020) The emerging potential of cold atmospheric plasma in skin biology, *Free Radical Biology and Medicine*, 161, 290-304.
4. Saadati, F., Mahdikia, H., Abbaszadeh, H.-A., Abdollahifar, M.-A., Khoramgah, M. S., Shokri, B. (2018) Comparison of Direct and Indirect cold atmospheric-pressure plasma methods in the B16F10 melanoma cancer cells treatment, *Scientific Reports*, 8, 7689.

Acknowledgments: This project was funded by Institut National du Cancer PLBIO022-117 (INCA_16714) and Institut des Sciences at Université Clermont Auvergne (IDS, UCA).

Fundamentals of Plasma Disinfection for the Inactivation of Viruses

M. Shaban[1,2*], A. P. Weber[2], N. Merkert[1]
[1] Institute of Applied Mechanics, Clausthal University of Technology, Clausthal-Zellerfeld, Germany
[2] Institute of Particle Technology, Clausthal University of Technology, Clausthal-Zellerfeld, Germany

Abstract:

Cold atmospheric plasma (CAP), used in biomedical applications like wound healing, cancer therapy, and sterilization, contains reactive oxygen and nitrogen species (RONS) that are effective in interacting with biological systems and inactivating viruses in aerosols. Our study explores the chemistry, stability, and reactivity of plasma-generated RONS. Using UV-vis, FTIR, and mass spectroscopy, we analyzed the concentration and decay of these species, and employed a radical-sensitive tracer reaction for deeper insights into their lifespan and reactivity. The experimental setup includes a cylindrical dielectric barrier discharge (DBD) with a disc-shaped inner electrode and an outer electrode, using a quartz glass tube both as a dielectric and for gas flow [1]. Various gas compositions and humidity levels were experimented with, combining parts of gas flow through a washing bottle with dry gas.

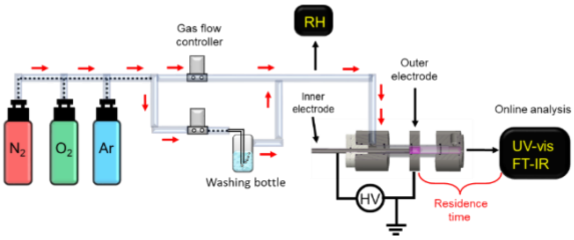


Figure 1: Schematic setup to produce DBD plasma species and control the plasma chemistry of DBD

FTIR spectroscopy in the spectral range from 800 to 2200 cm^{-1} was used to identify long living infrared active components produced by the air plasma jet. The short-living species such as OH were analysed using UV-vis spectroscopy of the DBD plasma and a radical sensitive tracer reaction [2].

Keywords: plasma disinfection, molecular dynamics (MD) simulations, dielectric barrier discharge (DBD), reactive oxygen and nitrogen species (RONS), plasma chemistry

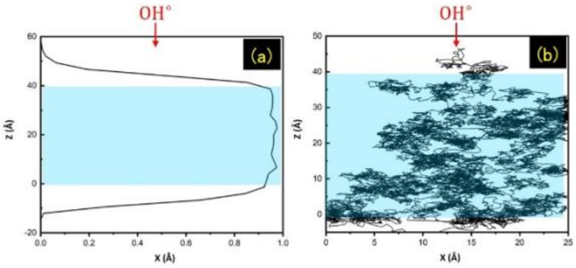


Figure 2: (a) Mass density profile of water slap; (b) trajectories of incident OH species in water. OH radical is impacting from the top of the water slab.

Molecular dynamics (MD) simulations, utilizing the LAMMPS software, were conducted alongside experimental work to explore how reactive plasma species like OH and H_2O_2 interact with liquid films enveloping viruses or bacteria in aerosols. These simulations aimed to understand if these species could penetrate the liquid layer to reach biomolecules. Our MD study involved placing plasma species at a distance above water and analyzing their interactions at 300 K. Findings indicate that OH radicals can effectively penetrate liquid layers up to 40 Å thick, suggesting their potential to reach and react with biological targets."

References:

1. Post, P., Jidenko, N., Weber, A. P., & Borra, J. P. (2016). Post-plasma SiOx coatings of metal and metal oxide nanoparticles for enhanced thermal stability and tunable photoactivity applications. Nanomaterials, 6(5), 91.
2. Shaban, M., Poostforooshan, J., & Weber, A. P. (2017). Surface-initiated polymerization on unmodified inorganic semiconductor nanoparticles via surfactant-free aerosol-based synthesis toward core–shell nanohybrids with a tunable shell thickness. Journal of Materials Chemistry A, 5(35), 18651-18663.

Cold Plasma Induced Enhancement of Recombinant Thaumatin Bioproduction

N. Parsekar, S. Akkermans, D. Kozak and J. F. M. Van Impe
BioTeC+ - Chemical & Biochemical Process Technology and Control,
Department of Chemical Engineering, KU Leuven, Gebroeders De Smetstraat 1, Ghent, Belgium

Abstract:

Heterologous protein expression in yeast has gained significant attention since its discovery in the late 19th century.[1] Most experimental approaches to enhance bioproduction are based on changing fermentation conditions such as pH, medium composition, aeration level, etc. Cold plasma has emerged as a valuable modality in bioproduction due to is non-thermal nature and can regulate the fermentation conditions without being invasive.[2] This study reveals the effects of sublethal exposure to helium cold plasma jet on the production of heterologous protein, thaumatin, by a genetically modified strain of methylotrophic *Pichia pastoris* GS115. A steady stream of helium plasma jet was obtained at 15 kHz and a gas flow rate of 10 L/min. The production medium containing methanol as substrate for thaumatin expression was treated in wide-mouth flasks for 2, 4, 6 and 8 min in quadruplicate before inoculation. The same duration of treatment followed once a day on the next three days. While no significant difference in thaumatin concentration was found between the individual treatment durations, comparison between treated and untreated groups showed a significant 16.7 % increase ($p = 3.9 \cdot 10^{-8}$) in thaumatin concentrations after the final treatment (Figure 1). The residual methanol levels were 11 % lower for the treated cultures after the final treatments of all durations, which provided a favorable environment for the cells to express the recombinant protein. Although methanol is a crucial substrate for heterologous protein expression in *P. pastoris*, concentrations beyond optimal range may lead to toxic conditions for the cells. This may result in the redistribution of pathways away from the protein of interest. To locate the exact effect of treatments, cell-free production medium containing methanol was given single helium jet plasma treatments for 8, 20 and 40 min in triplicate. This screening showed a 14.2, 21.3 and 32.5 % decrease in methanol concentrations as a function of plasma treatment indicating substrate degeneration or evaporation. Owing to the helium flow rate required to generate the plasma, a substantial reduction of volume (4.9, 8.4 and 11.2 %) and mass (7.0, 9.0 and 12,9 %) was recorded revealing the evaporation effect during the treatments. The findings of this study thus suggest that the increase in thaumatin levels is on account of a complex interplay between numerous effects occurring simultaneously. Evaporation of medium containing volatile methanol will inadvertently raise the apparent concentrations of thaumatin. The results of this study indicate that the impact of helium cold plasma jet via methanol degeneration is relatively higher than that due to methanol or liquid evaporation. While methanol breakdown may assist cells in the uptake of the substrate, it could also provide a more favorable environment to the cells by preventing methanol accumulation. This study thus demonstrates that cold plasma significantly modifies cellular environment through biochemical and thermodynamic changes conducive to enhanced bioproduction.

Keywords: helium plasma, plasma jet, bioproduction, *Pichia pastoris*, methylotrophic, thaumatin, methanol, heterologous protein.

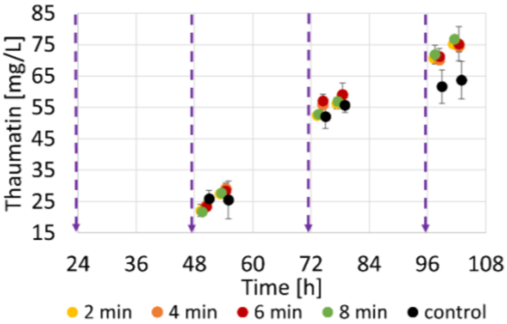


Figure 1: Thaumatin concentrations of day 3, 4 and 5 sampled two and six hours after 2-, 4-, 6- and 8-minutes treatments (↓) on each day.

References:

1. Cereghino, J. L., & Cregg, J. M. (2000). Heterologous protein expression in the methylotrophic yeast Pichia pastoris. FEMS Microbiology Reviews, 24(1), 45–66.
2. Farasat, M., Arjmand, S., Ranaei Siadat, S. O., et al. (2018). The effect of non-thermal atmospheric plasma on the production and activity of recombinant phytase enzyme. Scientific Reports, 8(1), 16647.

In vitro disinfection of MRSA biofilms with cold atmospheric plasma. Potential approach for hospital-acquired infection management

M. Lunder[1], S. Dahle[2], R. Fink[1]
[1] University of Ljubljana, Faculty of Health Sciences, Ljubljana, Slovenia
[2] University of Ljubljana, Biotechnical Faculty, Ljubljana, Slovenia

Abstract:
With bacteria becoming increasingly resistant to traditional antimicrobial agents, new approaches need to be explored. This study aimed to evaluate the efficacy of cold atmospheric plasma for managing methicillin-resistant *Staphylococcus aureus*. Resistant and susceptible *S. aureus* biofilms at 24, 48, and 72 hours were subjected to plasma treatments lasting 60, 120, and 180 seconds. Increasing the treatment duration led to a significant rise in cell reduction for both susceptible and resistant *S. aureus* ($p < 0.05$). We have demonstrated a reduction of > 5 CFU/cm^{-2} in 180 s plasma treatment. Additionally, the results indicated a substantial impact of plasma on cell metabolism and cell membrane integrity. Furthermore, we have not proved plasma can reduce biofilm biomass in the case of younger biofilms, although the 3-day-old biofilm was more susceptible, and its biomass was decreased. Furthermore, the study identified an accumulation of intrabacterial reactive oxygen species, confirming plasma's induction of oxidative stress. We have shown that continuous plasma exposure of bacterial cells does not cause resistance. The findings suggest that cold atmospheric plasma holds promise for the treatment of *S. aureus* and MRSA biofilms, offering a potential solution to the impending bacterial resistance crisis.

Keywords: cold atmospheric plasma, *S. aureus*, MRSA, reduction, metabolic activity, resistance.

References:

1. Yadav, B., & Roopesh, M. S. (2023). In-Package Atmospheric Cold Plasma Treatment and Storage Effects on Membrane Integrity, Oxidative Stress, and Esterase Activity of Listeria monocytogenes. Microorganisms, 11(3). https://doi.org/10.3390/microorganisms11030682.
2. Weltmann, K. D., Kinde, E., Von Woedtke, T., Hähnel, M., Stieber, M., & Brandenburg, R. (2010). Atmospheric-pressure plasma sources: Prospective tools for plasma medicine. Pure and Applied Chemistry, 82(6), 1223–1237. https://doi.org/10.1351/PAC-CON-09-10-35.
3. Silva, V., Araújo, S., Monteiro, A., Eira, J., Pereira, J. E., Maltez, L., Igrejas, G., Lemsaddek, T. S., & Poeta, P. (2023). Staphylococcus aureus and MRSA in Livestock: Antimicrobial Resistance and Genetic Lineages. Microorganisms, 11(1). MDPI. https://doi.org/10.3390/microorganisms11010124.

SICT 2024 / Plasma Tech 2024 Joint Session II. C: Plasma fundamentals / Modelling / Atomic and Molecular Processes Plasma Processing / Materials Interactions / Coatings

EUV Generated Hydrogen Plasmas

Tugba Piskin and Mark J. Kushner
Electrical Engineering &Computer Science, University of Michigan, Ann Arbor, MI 48109-2122 USA
tugbap@umich.edu, mjkush@umich.edu

Abstract:

The deployment of extreme ultraviolet (EUV) lithography systems is enabling fabrication of devices having nm critical sizes in microelectronic processing [1]. In one commercial system, the EUV photon beams are produced by ablating and ionizing tin droplets with a pulsed laser [2]. The ionized tin species emit photons having a 13.5 nm wavelength which are collected and directed by reflective optics and transferred to the scanner unit. The collector mirror is exposed to the tin plasma and may be coated by tin, which potentially results in a decrease in reflectivity and lifetime. Flowing hydrogen gas through the EUV chamber can help address this problem as an in-situ cleaning technique of deposited tin on the mirror. The hydrogen gas slows down fast tin radicals produced by the ablation while photoionization of the H_2 by the EUV produces a plasma [3]. The plasma produced H radicals can then etch the deposited tin on the mirror by producing stannane.

EUV photons with a 92 eV energy are capable of photo-dissociating and photo-ionizing hydrogen gas while producing energetic electrons and H atoms. Ions, energetic electrons, and photons in turn produce secondary electron emission from the surfaces, the latter of which can also produce energetic electrons. In this presentation, we discuss results from a computational investigation of EUV-generated hydrogen plasmas in an idealized lithography tool, including the etching of tin from mirror surfaces by hydrogen radicals. These investigations were performed with the Hybrid Plasma Equipment Model (HPEM) [4]. A surface reaction mechanism was developed to predict etch rates of tin from surfaces by radicals produced by the EUV plasma.

Mechanisms for plasma production, including electron energy distributions produced by photo-electrons, will be discussed. The fluxes, and energy and angular distributions of hydrogen radicals and ions to bounding surfaces and optics, and implied tin etch rates will be discussed for H_2 pressures of tens of mTorr to a afew Torr, and hundreds of Watts EUV power. A small bias applied to the substrate under the collector mirror to regulate the ion flux will be discussed as a means to non-destructively accelerate tin etch rates.

Keywords: EUV produced plasmas, lithography, plasma etching, photoionization.

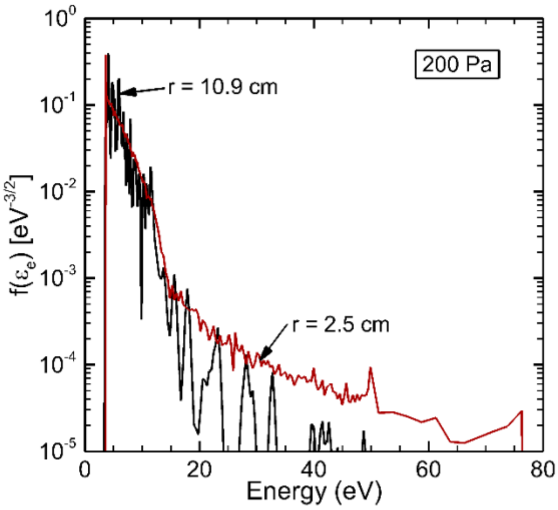


Figure 1: EUV photons (13.5 nm) produce energetic electrons upon photoionization of H_2. The electron energy distributions resulting from EUV generated photo-electrons in 200 Pa of H_2 are shown inside a lithography tool in the region illuminated by the EUV (r = 2.5 cm) and outside the region (r = 10.9 cm).

References:

1. H. J. Levinson, Jpn. J. Appl. Phys. **61,** SD0803 (2022).
2. J. Van Schoot, et al., "High-NA EUV lithography exposure tool: program progress", Proc. SPIE 11323, Extreme Ultraviolet (EUV) Lithography XI, 1132307 (23 March 2020); https://doi.org/10.1117/12.2551491
3. M. van de Kerkhof, et al., Rad. Effects Defects Solids **177,** 486 (2022).
4. T. Piskin et al., J. Appl. Phys. **133**, 173302 (2023).

Renewable electricity driven chemistry for energy conversion and storage: Novel pathways provided by plasma enhanced chemistry

Richard van de Sanden[1,2]

[1]DIFFER, P.O. Box 6336, 5600 HH Eindhoven, The Netherlands,
[2]Eindhoven Institute for Renewable Energy Systems (EIRES) and Department of Applied Physics,
Eindhoven University of Technology, Eindhoven, The Netherlands

Abstract

In a circular CO2 neutral society, the direct electrification of chemical conversion processes is one of the biggest challenges to achieve deep decarbonisation after 2030. The re-use of (air captured) carbon dioxide, the end product of the oxidation of dense energy carriers, for synthetic fuels and chemicals is required. Also in this context, nitrogen fixation in the form of NH3 or NOx is unquestionably one of the most important chemical conversion processes. These electricity driven chemical conversion processes are also needed to mitigate intermittency of renewable energy sources by providing seasonal storage, as well as non-fossil based feedstock for the chemical industry.

The use of electrons, from renewable electricity, or photons, directly from the sun, provide scientific and technological opportunities to develop novel pathways for chemical conversion. In this talk, after an introduction to the challenges facing the world in the next decades, I will discuss the opportunities of using plasmas, powered by renewable electricity, for scalable gas conversion of stable key molecules such as CO2 and N2. In particular, I will address the use of microwave generated plasma to dissociate CO2 into CO and O2, the formation of NOx from N2/O2 mixtures and the possible, often claimed, role of nonequilibrium vibrational kinetics in these conversion processes. An outlook on potential gamechanging application of non-thermal plasma, next to the use as a high temperature heating source, and in combination with catalyst, will be discussed.

Multi-Scale Model for High Aspect Ratio TiN Etching in a Cl_2/Ar Inductively Coupled Plasma

Lado Filipovic * and Tobias Reiter
CDL for Multi-Scale Process Modeling of Semiconductor Devices and Sensors at the
Institute for Microelectronics, TU Wien, Gußhausstraße 27-29/E360, 1040 Vienna, Austria

Abstract:

The realization of plasmonic sensors with complementary metal-oxide semiconductor (CMOS) fabrication relies on using Si-compatible materials, such as TiN [1]. This requires the generation of high aspect ratio (HAR) TiN trenches which can be formed by etching in a Cl_2/Ar plasma with high selectivity over SiO_2 [2]. To optimize the fabrication process, simulations can be performed; however, there is a large discrepancy in the size scale when simulating plasma reactor conditions (cm-scale) and the nanosized trench features (nm-scale). At the feature scale, the main model parameters which vary under changing reactor conditions are the flux (Γ) of arriving radicals, ions, and passivating species, and their sticking probabilities (β). At the reactor scale, however, the model parameters typically include the chamber pressure (p), temperature (T), the applied power (P), the concentration ratios of the individual gas molecules (C_{Cl_2} and C_{Ar}), and the total gas flow rate (Q_{sccm}).

We perform thorough reactor-scale simulations using the finite element method (FEM) to extract the impact of each parameter on the arriving Cl radical flux Γ_{Cl} on the wafer since etching proceeds when Cl radicals react with Ti and N to form $TiCl_x$ and NCl_x products. A multi-variable spline interpolation is then applied on a regular grid to link the reactor-scale parameters to Γ_{Cl}. The sticking probability is calculated from experimentally observed flat-wafer etch rates and is a function of the reactor pressure and temperature:

$$\beta = \frac{r_{\mathrm{TiN}}\rho_{TiN}}{M_{\mathrm{TiN}}p}\sqrt{2RTM_{\mathrm{Cl}}}\ ,$$

with r_{TiN} and ρ_{TiN} the etch rate and molar density of TiN, R the universal gas constant and M_{X} the molar mass of species X. From the experiments performed in [3] and ignoring ion etching a sticking probability of 0.00975 moles per second is calculated. At the feature scale, we also include the thermal desorption of Cl species using an evaporation flux with an activation energy for desorption of 0.5 eV.

For the reactor-scale simulations, an inductively coupled plasma (ICP) is generated by 13.56 MHz rf power fed through a multi-turn coil on the dielectric window. The feed gases are supplied through an inlet ring located under the dielectric window, while they are pumped away through a vacuum port at the bottom. The electron momentum transfer cross sections are obtained from [4][5]. At the feature scale, we use our in-house developed process simulator ViennaPS [6] and apply a single-particle chemical etch model with the flux and sticking coefficient found from the reactor simulations described above. The set of reactor simulations are only performed once to design the multi-variable spline interpolation model (see Figure 1), which provides the flux and sticking coefficients at the top of the waver or feature. We then apply Monte Carlo ray tracing to calculate the resulting fluxes and surface etch rates in complex nanosized features, such as HAR trenches and holes.

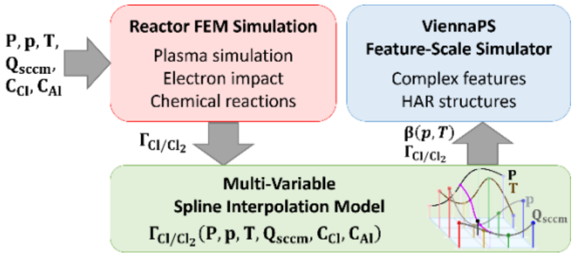


Figure 1: Implementation of a multi-scale plasma etch model using multi-variable spline interpolation.

Keywords: plasma etching, TiN, Cl_2/Ar, multi-scale simulations, feature-scale model

References:

1. Hamza, M.E., Othman, M.A., Swillam, M.A. (2022) Plasmonic Biosensors: Review, *Biology*, 11(5), 621. doi: 10.3390/biology11050621
2. Kim, D.-P. et al. (2011) Dry Etching of TiN in N_2/Cl_2/Ar adaptively coupled plasma, *Vacuum*, 86(4), pp. 380-385. doi: 10.1016/j.vacuum.2011.08.002
3. Woo, J.-C., Kim, S.-H., Kim, C.-I. (2011) Etch Characteristics of TiN/Al_2O_3 Thin Film by Using a Cl_2/Ar Adaptive Coupled Plasma, *Vacuum*, 86(4), pp. 403-408. doi: 10.1016/j.vacuum.2011.08.006
4. Carbone, E. et al. (2022) Data Needs for Modeling Low-Temperature Non-Equilibrium Plasmas, *Atoms*, 9(1), 16. doi: 10.3390/atoms9010016
5. SIGLO database, www.lxcat.net, retrieved on December 11, 2023.
6. ViennaPS (2023), https://github.com/ViennaTools/ViennaPS, accessed on December 12, 2023.

Simple Plasma-triggered Reduction & Exfoliation of Graphene Oxide and Fabrication of 2D and 3D rGO Structures

Richard Krumpolec [1,*], František Zelenák [1], Jana Jurmanová [1], Monika Stupavská [1], Michal Pazderka [1], Zdeněk Moravec [2], Mirko Černák [1]
[1] Department of Plasma Physics and Technology, CEPLANT—R&D Center for Plasma and Nanotechnology Surface Modifications, Faculty of Science, Masaryk University, Brno, Czech Republic
[2] Department of Chemistry, Faculty of Science, Masaryk University, Brno, Czech Republic

Abstract:
A top-down approach by oxidation of graphite to graphene oxide (GO) and its reduction via various techniques gained popularity for a mass production of thin conductive graphene-like films. The most advantageous alternative to pure graphene is reduced graphene oxide (rGO) as it is almost alike graphene in terms of properties and can be fabricated from GO using several reduction/exfoliation techniques. The known reduction/exfoliation techniques, however, have limitations such as high levels of toxicity, hazardousness, risk of explosions, and high time and energy consumption. The complicated reduction/exfoliation of GO become a major bottleneck that restricts the large-scale production of rGO. The research team of CEPLANT have developed a simple, fast, low-cost and chemical-free fabrication method of 2D and 3D rGO structures using an electrical plasma-triggered reduction&exfoliation of 3D porous GO aerogel-like materials [1] and also 2D GO paper-like films [2] starting at temperatures not higher than 100 °C.

The direct exposure of self fabricated GO structures to low-temperature atmospheric plasma generated by the Diffuse Coplanar Surface Dielectric Barrier Discharge (DCSBD) in technical nitrogen or standard air, triggers the self-propagating reduction&exfoliation modification of GO into the rGO (Figure 1). The rapid modification of GO into rGO do not result in abrupt disintegration of the sample and led in an approximately 10^5-fold decrease of the rGO resistivity compared to the original GO.

The fast plasma-triggered process of GO reduction&exfoliation will be compared with the heat treatment and laser induced reduction of the same GO structures.

This novel plasma-triggered reduction&exfoliation method opens new opportunities for low-cost industrial-scale production of conductive rGO and rGO-based composites for various applications. We will will present the results of plasma and plasma-chemical functionalization of rGO.

Finally, we will show how fabricated rGO-based structures can be tailored for particular applications in the field of i) electromagnetic shielding; ii) energy storage, and iii) water treatment.

Keywords: atmospheric plasma, DCSBD, low-temperature, graphene oxide, reduced graphene oxide, self-propagating reduction, exfoliation, laser-treatment, heat-treatment, applications.

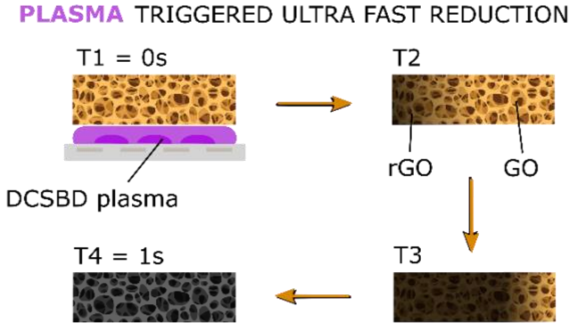


Figure 1: Figure illustrating the process of fast DCSBD plasma-triggered self-propagating reduction and exfoliation of graphene oxide into reduced graphene oxide.

References:

1. Krumpolec, R., et al. (2022), High conductive rGO sheets fabricated by mild, low-cost and scalable plasma-triggered reduction-exfoliation of 3D aerogel-like graphene oxide, *FlatChem*, 35, 100403.
2. Zelenák, F., et al. (2023), Fast, scalable, and environmentally friendly method for production of stand-alone ultrathin reduced graphene oxide paper, *Carbon*, 215, 118436.

Non-Thermal Plasma Synthesis of Imine Macrocycles

Patrycja Roszkowska[1]†, Abbie Scholes[1]†, James L. Walsh[2], Timothy L. Easun[3], Anna G. Slater[1]
[1] Department of Chemistry and Materials Innovation Factory, School of Physical Sciences, University of Liverpool, UK;
[2] Engineering and Technology, School of Physics, University of York, UK
[3] School of Chemistry, Haworth Building, University of Birmingham, UK

Abstract:

Plasma-liquid interactions are known for being intricate systems. Non- thermal plasma (NTP) in contact with solutions comprised of solvent and reagents can react with both, leading to complex reactive mixtures of short- and long-lived species, that are difficult to predict and control. [1]

Nonetheless the growing interest in using plasma for chemical reactions that are done in solutions is observed. NTP systems offers unique properties, mild reaction conditions and highly energised environments already confirm importance, especially in radical driven reactions.

For instance, NTP in contact with water was used to initiate radical reactions to dehalogenate iodo-substituted benzoates, with high yields and mild conditions. [2] Another example with NTP interaction with aromatic ring compounds was amination of benzene. [3], [4]

NTP-liquid systems have been extended to organic and hybrid materials: metal-organic [5] and covalent organic frameworks (COFs) [6] have been formed using NTP. The latter example demonstrates the potential of NTP to accelerate acid-catalysed reactions that form stable solid products, such as the formation of boronate ester, boroxine, and Schiff base COFs, but this has not yet been demonstrated for discrete molecular species such as macrocycles.

We explore the use of Non-Thermal Plasma (NTP) as an efficient method for the synthesis of imine macrocycles. Macrocycles are candidates for wide-ranging applications, yet their synthesis is often low-yielding, and can be poorly reproducible and resource-intensive, limiting their use.

We discovered that NTP-mediated macrocyclisations consistently achieved high yields of up to 97 % in reduced reaction times compared to the standard non-plasma method, and removed the need for purification across a range of different aldehyde substrates.

† Roszkowska and Scholes contributed equally to this work

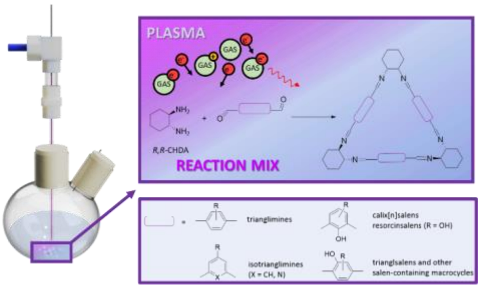


Figure 1: Graphical abstract illustrating reaction setup and the schematic representation of imine macrocycle synthesis

Keywords: non-thermal plsama, chemical engineering, non-thermal plasma in contact with liquid, macrocycle, imine condensation.

References:

1. V. V. Kovačević, G. B. Sretenović, B. M. Obradović, and M. M. Kuraica, ‘Low-temperature plasmas in contact with liquids—a review of recent progress and challenges’, *J Phys D Appl Phys*, vol. 55, no. 47, p. 473002, Nov. 2022
2. Y. Gorbanev, D. Leifert, A. Studer, D. O’Connell, and V. Chechik, ‘Initiating radical reactions with non-thermal plasmas’, *Chemical Communications*, vol. 53, no. 26, pp. 3685–3688, 2017
3. V. Dupont, S. Ognier, G. Morand, C. Ollivier, L. Fensterbank, and M. Tatoulian, ‘Direct Amination of Benzene with Ammonia by Flow Plasma Chemistry’, *Chemistry - A European Journal*, vol. 29, no. 56, pp. 1–8, 2023
4. X. Xu, X. Zhao, J. Tang, Y. Duan, and Y. Tian, ‘Direct Amination of Benzene with Molecular Nitrogen Enabled by Plasma-Liquid Interactions’, *Angewandte Chemie International Edition*, vol. 61, no. 24, Jun. 2022
5. Q. Wei *et al.*, ‘Plasma Meets MOFs: Synthesis, Modifications, and Functionalities’, *The Chemical Record*, vol. 202200263, Jan. 2023
6. J. He *et al.*, ‘Low Power, Low Temperature and Atmospheric Pressure Plasma-Induced Polymerization: Facile Synthesis and Crystal Regulation of Covalent Organic Frameworks’, *Angewandte Chemie - International Edition*, vol. 60, no. 18, pp. 9984–9989, 2021

Plasma Diagnostics for Ion Energy and Flux measurements in Etching and Deposition processes

A. Verma, T. Gilmore
Impedans Ltd, Chase House, City Junction Business Park, Northern Cross, Dublin, D17 AK63, Ireland

Abstract:

Plasma processes are used extensively in deposition and etching operations used in the fabrication of integrated circuits (ICs). These applications have led to the development of various kinds of plasma diagnostic tools. The diagnostics used in any plasma assisted process serve twofold. Firstly, they are used to understand the physics and chemistry of the underlying process and help to optimize the process and hardware output. Secondly, they serve as sensors for process control. For this purpose, the diagnostics must be robust, repeatable and versatile to suit diverse plasma systems used for processing of various metals, ceramics and polymer materials. Our focus is on the plasma assisted etching and deposition process for nanoscale fabrications. As the energy and angular distributions of incoming ions can be varied in principle, and this makes it possible to achieve improved filling of features such as trenches and vias. It is also possible to alter film properties such as stress, density and composition by changing process conditions.

Impedans offers fully automated advanced Retarding Field Energy Analyzers (RFEAs) for ion energy and ion flux measurements in plasma [1, 2]. The *Semion RFEA* measures the ion energies hitting a surface, the ion flux, negative ions and bias voltage at any position inside a plasma chamber using an array of integrated sensors. This can measure in RF, Pulsed DC and Tailored Waveform biased plasmas. On the other hand, the *Quantum system* is an energy resolving gridded quartz crystal microbalance (QCM), used to measure the ion-neutral fraction hitting a surface inside a plasma reactor. This instrument also measures the etching/deposition rate, ion energy, ion flux and bias voltage. It may also serve as a way to measure radical etch rates. Finally, the *Vertex* system is able to measure ion energy distributions functions for different aspect ratios. Some simple RFEA modifications can also allow ion angular distribution functions to be measures.

In this talk, we will highlight the successful measurements done by Impedans' advanced RFEA systems in various plasma applications enabling accurate and precise control of etching /deposition profiles on different materials and various plasma chemistries, with a focus on modern plasma industry challenges.

Keywords: Ion energy distribution function, ion flux, etching, deposition, plasma process, plasma diagnostics, retarding field ion energy analyzer.

References:

1. Impedans Ltd, Dublin, Ireland [www.impedans.com]
2. S. Sharma (2016), Ph.D. Thesis, Dublin City University.
3. H. B. Profijt et al. (2013), Substrate-biasing during plasma-assisted atomic layer deposition to tailor metal-oxide thin film growth, *J. Vac. Sci. Technol.*, A 31, 01A106
4. M. H Heyne et al. (2019), A route towards the fabrication of 2D heterostructures using atomic layer etching combined with selective conversion, *2D Mater.*, 6, 035030.
5. S. Karwal et al. (2020), Plasma-Assisted ALD of Highly Conductive HfN_x: On the Effect of Energetic Ions on Film Microstructure, *Plasma Chemistry and Plasma Processing* 40, 697–712.

The effect of plasma parameters on the surface treatment of electrocatalysts

He Li, Christian Schulze, Jan Benedikt
Institute for Experimental and Applied Physics, Kiel University, Kiel, Germany

Abstract

Plasma technology is of vital importance in the research field of nanomaterials which offers a green and efficient process for functionalization and treatment of surfaces. Most of the previous researches focus on the process parameters, but the relationship between plasma properties, such as ion energy or fluxes of reactive species, and materials properties is still unclear. In this work, a new reactor was assembled which is combining ICP plasma with DC bias to control the plasma density and ion energy separately during the treatment, and transition metal oxides were used as the substrate to compare the electrochemical performance before and after plasma treatment. The plasma parameters during treatment were measured by the energy-resolved ion mass spectrometry which were analyzed with the corresponding catalytic performances to optimize the treatment process and fundamentally understand the reaction processes and mechanisms between plasma and material surfaces, thus providing a theoretical basis for the future application in the material researches.

Surface Functionalization of Industrial Materials via PECVD and its Influence on Oxygen Nucleation

Julian Heinrich[1,2], Karin Schwarzenberger[1,2], Xuegeng Yang[1,2], Kerstin Eckert[1,2]
[1] Institute of Fluid Dynamics, Helmholtz-Zentrum Dresden-Rossendorf, 01328 Dresden, Germany
[2] Institute of Process Engineering and Environmental Technology, Technische Universität Dresden, 01069 Dresden, Germany

Abstract:
Proton-exchange membrane (PEM) electrolysis is one of the mostly used principles for H_2 generation. To increase its profitability and the attractiveness of ‘power-to-gas’ strategies, an enhanced O_2 separation in the anodic cycle is necessary to reduce overvoltage and improve cooling efficiency. In this work, we investigate the surface functionalization by plasma-enhanced chemical vapour deposition (PECVD) of stainless steel and polypropylene to tune the affinity of the nonpolar gas to the materials by varying their hydrophilicity/hydrophobicity.

Hereby, hexamethyldisiloxane (HMDSO) and 1H,1H,2H,2H-perfluorooctylacrylate are used as monomers for the polymerization-based plasma coating process. The resulting surface properties are characterized via water contact angle measurement. We observed that the stability of the coating, its aging and the general effect on the wettability are highly dependent on the surrounding media. Our experimental findings are supported by XPS to determine the elemental composition and changes of the substrate surfaces. Via optical recordings we demonstrate and analyze the enhancing influence of hydrophobic surfaces on the O2 nucleation as well as the hindrance of nucleation by hydrophilic ones. The generated data provides first insights on how the oxygen separation of the anodic cycle of PEM electrolyzers can be tuned by surface functionalization via PECVD.

Keywords: surface functionalization, PECVD, wettability, oxygen, bubble nucleation

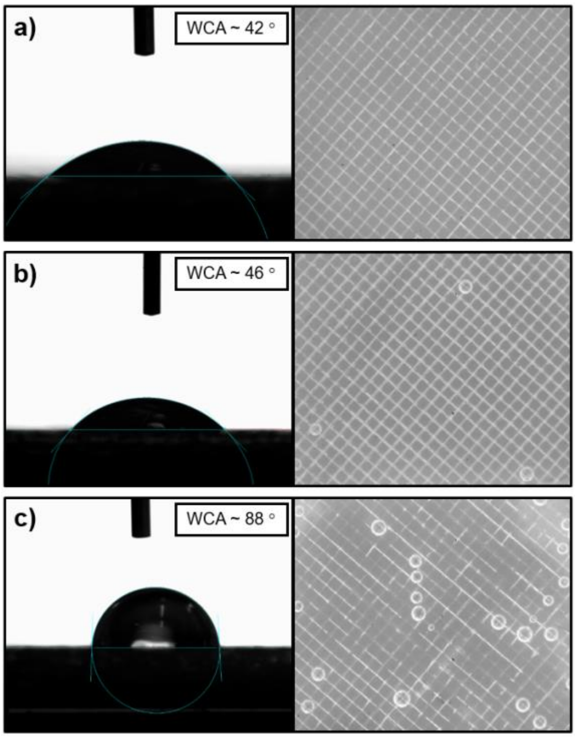


Figure 1: Water contact angle measurements and oxygen bubble nucleation behaviour on a) HMDSO-funtionalized polypropylene, b) untreated polypropylene and c) 1H,1H,2H,2H-perfluorooctylacrylate-functionalized polypropylene.

Atmospheric Pressure cold Plasma processing of textiles – applications in functional finishings

Shital Subhash Palaskar
Plasma Research Laboratory, The Bombay Textile Research Association
Lal Bahadur Shastri Marg, Ghatkopar (West), Mumbai 400086, Maharashtra, India

Abstract:

The Greige textile goods need to be converted in the finished product to make it comfortable to wear, attractive and feel good. The process of converting greige textile into finished fabrics is called wet processing. Classical textile wet processing techniques are chemical, water and energy intensive. Therefore, to make it non-aqueous, environmental friendly Plasma processing is an economical and green alternative and hence it is becoming increasingly popular. This Paper presents the functional finishing of textile materials using etching and deposition process of low temperature atmospheric pressure plasma technology.

Plasma can be explained as the fourth state of matter where the constituents of gas atoms can be activated by addition of energy to produce highly energetic electrons, protons, neutrons, and radicals. The bombardment of these energetic particles result in cleavage of the chemical covalent bonds thereby giving rise to physico-chemical surface modification. The extent of physico-chemical surface modification of the textile material is dependent on the various plasma processing parameters including the power of plasma generation, duration of the exposure, type of gases used etc. In this study, atmospheric pressure, dielectric barrier discharge (DBD) cold plasma is used for functional finishing of the various textile materials including, cotton, silk, polyester, nylon, polypropylene etc.

for improving performance such as improved color up-take, enhancing adhesion with polymer coated fabrics, imparting functionalities like hydrophilicity, hydrophobicity, antibacterial antistatic, electrical conductivity, Multifunctional finish with superior wash durability compared to classical functional finishes is reported.

One of the functionality imparted through plasma processing on the nylon fabric is depicted in the figure 1.

Keywords: Surface Etching, Plasma enhanced chemical vapour deposition (PECVD), hydrophilicity, hydrophobicity, antibacterial, antistatic, Multifunctional, durable

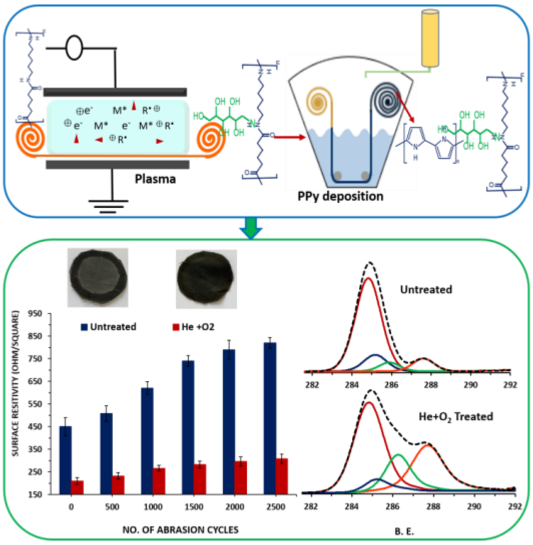


Figure 1: Figure illustrating the fundamentals of plasma reaction with the textile substrate to impart the conducting properties and durability study. It also represents the generation of functional groups after plasma as detected by XPS analysis. reference 2.

References:

1. Shital S. Palaskar (2023) Adhesion studies of atmospheric pressure plasma-modified polyester fabrics with polyurethane coating, Journal of Adhesion Science and Technology, 37:23, 3468-3484, DOI: 10.1080/01694243.2023.2202951
2. Smita Deogaonkar-Baride, Shital S. Palaskar (2022) Atmospheric pressure plasma treatment for enhancing the conducting properties of polypyrrole coated nylon fabric, Journal of Applied Polyer Science, 139,26, e52443, doi.org/10.1002/app.52443
3. S.S. Palaskar, A.N. Desai & S.R. Shukla (2016) Development of multifunctional cotton fabric using atmospheric pressure plasma and nano-finishing, The Journal of The Textile Institute, 107:3, 405412, DOI: 10.1080/00405000.2015.1034932
4. Kale KH, Palaskar S. Atmospheric pressure plasma polymerization of hexamethyldisiloxane for imparting water repellency to cotton fabric. Textile Research Journal. 2011;81(6):608-620. doi:10.1177/0040517510385176
5. **https://scholar.google.com/citations?user=TjWX0EsAAAAJ&hl=en**

Enhancing adhesion of recycled and non-recycled EVA via atmospheric pressure plasma treatments

C. Ruzafa-Silvestre*, V.M. Serrano-Martínez, M.D. Romero-Sánchez, E.Orgilés-Calpena
[1]INESCOP. Footwear Technology Centre, Campo Alto Campground, 03600 Alicante, Spain
* Correspondence: cruzafa@inescop.es

Abstract:

This investigation evaluates atmospheric pressure plasma treatments (APT) for improving the adhesion of ethylene-vinyl acetate (EVA) to leather using reactive polyurethane hot melt (HMPUR) adhesive, focusing on both recycled and non-recycled EVA [1]. The study sought to enhance adhesion by modifying surface properties through APT, employing optimisation of working parameters (nozzle distance and speed) and various characterisation techniques including Fourier-Transform Infrared Spectroscopy (FTIR), X-ray Photoelectron Spectroscopy (XPS), Scanning Electron Microscopy (SEM), roughness and contact angle measurements, Surface Free Energy (SFE) evaluations, and T-peel strength tests. These methods assessed the treatments' effects on EVA's chemistry, morphology, and adhesion.

Comparative analysis of untreated, chemically treated (less eco-friendly methods like primer application), and plasma-treated samples revealed that APT significantly improves surface properties, enhancing adhesive bonding. This enhancement is credited to changes in chemical composition, increased mechanical interlocking, and better wettability, which collectively result in stronger bonds as evidenced by T-peel tests.

The research concludes that APT is an effective and sustainable technique to promote the use of recycled materials in high-demand adhesive contexts. Advocating for the integration of plasma treatments, the study highlights their potential to boost the sustainability and performance of adhesive joints based on EVA and leather, especially relevant to the fashion and footwear sectors [2]. This aligns with increasing demands for eco-friendly and durable products, pushing sustainable manufacturing forward. The findings underscore the broad applicability of plasma treatments and the need for further research to optimise these processes and fully understand their environmental impacts [3].

Keywords: EVA, leather, atmospheric pressure plasma treatments, surface modification, adhesive bonding, fashion, sustainability

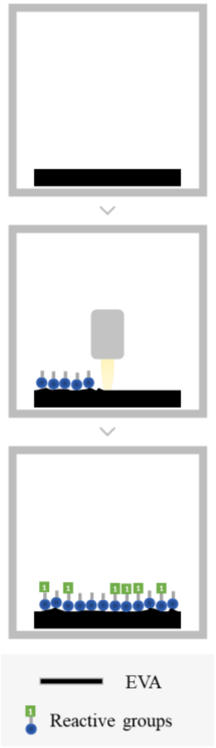


Figure 1: Sequential illustration of atmospheric pressure plasma treatment on EVA material.

References:

1. Iqbal, M., Dinh, D. K., Abbas, Q., Imran, M., Sattar, H., Ul Ahmad, A. (2019), Controlled Surface Wettability by Plasma Polymer Surface Modification, Surfaces., 2(2), 349-371.
2. Ruzafa-Silvestre, C., Serrano-Martínez, V. M., Arán-Ais, F., Orgilés-Calpena, E. (2024), Durability of the effectiveness of atmospheric plasma treatment applied to thermoplastic Styrene-Butadiene-Styrene (SBS) rubber, J. Adhesion., 1-27.
3. Levchenko, I., Xu, S., Baranov, O., Bazaka, O., Ivanova, E., Bazaka, K. (2021), Plasma and Polymers: Recent Progress and Trends, Molecules., 26(13), 4091.

Enhancing Cotton Fabrics Dyeability through Low-Pressure Plasma Activation Surface Treatment

V. M. Serrano Martínez [1,*], C. Ruzafa Silvestre [1], M. D. Romero Sánchez [1], E. Orgilés Calpena [1]
[1] Footwear Technology Centre, Campo Alto Campground, 03600 Alicante, Spain
* Correspondence: vmserrano@inescop.es

Abstract:

This innovative study addresses the enhancement of dyeing properties in cotton fabrics through surface activation treatment using low-pressure oxygen plasma. This technique focuses on modifying the cotton surface to increase its capacity for absorbing polar liquids and dyes, thereby overcoming challenges associated with conventional surface pre-treatments before dyeing [1].

The research commences with a plasma treatment, involving the exposure of cotton fabrics to an ionized oxygen environment, varying the operating time condition (from 30s to 120s). This process induces significant changes in the physical and chemical properties of the textile surfaces. Advanced analytical techniques such as Fourier Transform Infrared Spectroscopy (FTIR), X-ray Photoelectron Spectroscopy (XPS), and Scanning Electron Microscopy (SEM) were employed to evaluate these alterations. Analyses revealed modifications in chemical composition and surface morphology, resulting in increased hydrophilicity and enhanced dye affinity.

Experimental results demonstrated that treated cotton fabrics absorbed water and dyes more efficiently compared to untreated fabrics. This enhanced dye absorption leads to textiles with more vibrant colors and greater uniformity in color application. Furthermore, the study highlights the potential of plasma technology as a dry treatment technique, offering an eco-friendly substitute for traditional pre-dyeing processes, which typically consume large amounts of water and chemicals to prepare the surface for dyeing..

This innovative approach not only improves the quality and performance of cotton textiles but also promotes environmentally friendly manufacturing practices. The study paves the way for future research in the field of plasma technology and its application in the textile industry, offering promising prospects for the development of sustainable and efficient production processes [2].

Keywords: Low-Pressure Oxygen Plasma, Cotton Fabric, Surface Activation, Dyeing Enhancement, Textile Industry, Hydrophilicity, Surface Chemistry.

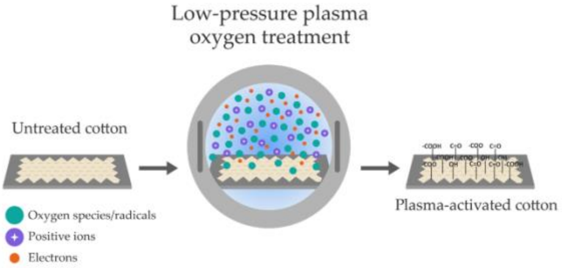


Figure 1: Illustrative sketching of the transformation of cotton fabric: from untreated to plasma-activated via low-pressure oxygen plasma treatment.

References:

1. Karahan, H.A.; Özdoğan, E. Improvements of Surface Functionality of Cotton Fibers by Atmospheric Plasma Treatment. *Fibers and Polymers* **2008**, *9*, 21–26, doi:10.1007/s12221-008-0004-6.
2. Gotmare V.D.; Samanta Kartick K.; Patil V.; Basak S.; Chattopdhayay S.K. Surface Modification of Cotton Textile Using Low-Temperature Plasma. *International Journal of Bioresource Science* **2015**, *2*, 37–45.

Industrially Applicable Atmospheric-Pressure Plasma Treatment of Nonwovens, Cotton and Electrospun Nanofibres for the Enhancement of Their Performance Properties in Various Areas of Applications

D. Kováčik [1, 2*], O. Galmiz [1], J. Kelar [1], L. Zahedi [1], S. Ben Hamida [1], V. Medvecká [2], R. Krumpolec [1], L. Mynářová [3], M. Černák [1]

[1] Department of Plasma Physics and Technology, CEPLANT – R&D Center for Plasma and Nanotechnology Surface Modifications, Faculty of Science, Masaryk University, Brno, Czech Republic

[2] Department of Experimental Physics, Faculty of Mathematics, Physics and Informatics, Comenius University in Bratislava, Slovak Republic

[3] NAFIGATE Corporation a.s., Prague, Czech Republic

Abstract:

Despite many significant advantages demonstrated through laboratory and industrial prototype plasma devices operating at low and atmospheric pressure, plasma treatment of textile materials on an industrial scale has not yet experienced significant growth. This situation is caused by the lack of applied research in the given area, the time-consuming development of suitable industrial plasma systems, and the late focus on developing in-line plasma sources operating atmospheric pressure. The discharge, which has the prerequisites for use in the textile industry works based on a dielectric barrier discharge and was named diffuse coplanar surface barrier discharge (DCSBD). It is characterized by generating a non-isothermal plasma in the ambient air in a thin layer (~0.3 mm) of high power density with a high representation of diffuse plasma, due to which it appears macroscopically homogeneous without any stabilization by the flowing gas or admixture of noble gases. [1] DCSBD is robust and easily scalable, capable of operation in dusty and humid environments, which predisposes it to direct industrial use in production lines for the treatment of textile and (nano-) fibre materials. In the CEPLANT centre, the DCSBD is successfully used in research to optimise the surface treatment of various materials, including textiles. It was successfully used for the permanent hydrophilic treatment of the surface of the lightweight polypropylene (PP) non-woven fabrics as well as their high-speed activation (450 m/min) directly on the production line before subsequent coating with surfactants. DCSBD plasma treatment of the non-woven fabric surface has been shown to be effective in improving the adhesion of water-based dyes and inks, immobilized antimicrobial active substances or in their lamination. Besides that, DCSBD plasma treatment can be used for surface activation of PP nonwoven, serving as a substrate for the electrospun nanofibre layer to enhance its adhesion but also for the treatment of the nanofibre layer itself to make it hydrophilic. These effects of plasma treatment can be utilized in the production of nanofibre membranes to enhance their water and air filtering performance properties. This was verified in the collaboration with NAFIGATE Corporation a.s. company under industrial conditions (Figure 1) within the ELIIT project. In connection with nanofibres, other research activity at the CEPLANT centre are also optimizing electrospun nanofibres for enhanced tissue engineering performance assisted by atmospheric pressure plasma and plasma-assisted calcination of inorganic nanofibres. DCSBD plasma was also used for enhancement of the dyeability and antibacterial properties of cotton fabric.

Keywords: atmospheric-pressure plasma, DCSBD, textile treatment, electrospun nanofibres, hydrophilicity, adhesion

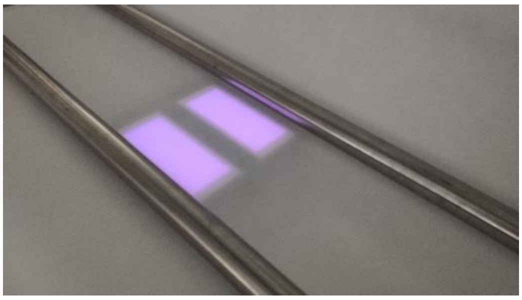


Figure 1: Photo illustrating the treatment using two DCSBD plasma units of the electrospun nanofibre layer on the bottom side of PP nonwoven carrier substrate in the production line.

References:

1. Černák, M., Kováčik, D., Ráheľ, J., *et al.* (2011), Generation of a high-density highly non-equilibrium air plasma for high-speed large-area flat surface processing, *Plasma Phys. Control. Fusion*, 53, 124031

High-Throughput Synthesis of Nanomaterials by Spatial-Temporal Control of Modulated Induction Thermal Plasma Fields and its Optimization by Machine Learning

Yasunori Tanaka[1*]
[1] Faculty of Electrical Engineering & Computer Science, Kanazawa University, Kanazawa, Japan

Abstract:

Nanoparticles/nanopowders are widely recognized for their unique physico-chemical properties, which have made them indispensable in various fields, including electronics, biomedicine, energy, and environmental applications. For example, silicon-related nanoparticles are attracting attention as a new anode material for Lithium-ion batteries (LIB), because Si has the theoretical capacity of 4.2 Ah/g, which is much larger than that of 0.37Ah/g of current graphite anodes. As new batteries, All-Solid-State Lithium-Ion Secondary Batteries (ASS-LISB) are also being developed with multi-composite nanoparticles. Metal oxide nanoparticles such as TiO_2 and ZnO with some metallic ion-doping are well known to induce efficient photocatalysis even by visible light. It is worth noting that efficient mass production methods have been developed to meet the growing demand for these materials.

We have been developing a new technique for synthesizing nanoparticles called the 'PMITP+TCFF method[1].' The method combines pulse-modulated induction thermal plasma (PMITP) with time-controlled feeding of feedstock (TCFF) to enhance synthesis efficiency. The PMITP maintains a thermal plasma field through coil current modulation, creating periodic variations between higher and lower-temperature plasma states. The TCFF synchronously and intermittently supplies feedstock to further improve the process. At 20 kW power, this method achieves impressive production rates, yielding various metallic ion-doped TiO_2 nanopowders [1] and Si nanoparticles/nanowires[2]. Building on these advancements, we have introduced the 'Tandem-MITP+TCFF method' to achieve even higher production rates[3]. This method involves two independent coils in a single plasma torch, generating a long axial thermal plasma field. The upper coil ensures stability, while the lower coil sustains a modulated plasma state. The Tandem-MITP+TCFF approach has demonstrated potential for high-rate nanoparticle production, with rates exceeding several hundred g/min in experimental settings.

It is widely acknowledged that efficient mass production of nanoparticles is crucial for their widespread use. However, optimizing the numerous parameters in the Tandem-MITP+TCFF method presents significant challenges that require careful consideration and collaboration among experts in the field. To address the issue at hand, we developed a numerical thermofluid model to simulate the thermal plasma field, feedstock particle behavior, and nanoparticle production and transport [4]. The model was validated against measured radiation intensity distribution and feedstock particle motions. Additionally, the study includes optimization of operating conditions using a numerical fluid dynamics model and machine learning techniques. The study utilized a method called Sequential Approximate Multi-Objective Optimization Method (SAMOO) with Radial Basis Function-Neural Network (RBF-ANN) [5,6] to optimize nine control parameters for synthesizing larger amounts of smaller Si nanoparticles. The developed numerical model and optimization techniques for tandem-MITP+TCFF method contribute to understanding and enhancing the controlled parameters in these methods. These advancements hold great promise for meeting the increasing demand for nanoparticles in various applications, further propelling the evolution of future technologies and addressing global challenges.

References:

1. Kodama K., et al., (2014) J. Phys. D: Appl. Phys. 47, 195304.
2. Ishisaka Y., et al. (2017), Applied Physics Express, 10, 096201
3. Furukawa R., et al, (2022) J.Phys.D:Appl.Phys. 55, 044001
4. Furukawa R., et al, (2021) Powder Technol., 389, 460
5. Kitayama S., et al., (2013) Strict Multidisc Optim., 48, 501
6. Tanaka Y., et al., (2023) Int.Symp.Plasma Chem. (ISPC-25)

Diffuse Dielectric Barrier Discharges at atmospheric pressure: Technical and Scientific locks for thin film deposition

Nicolas Naudé[1], Antoine Belinger[1], Hubert Caquineau[1], Simon Dap[1], Fiorenza Fanelli[2]

[1]LAPLACE, Université de Toulouse, CNRS, INPT, UPS, 31062 Toulouse, France
[2]National Research Council (CNR), Institute of Nanotechnology (NANOTEC), Bari Unit, Bari, Italy

Abstract:

Dielectric Barrier Discharges (DBDs) are a type of atmospheric pressure discharge that can be used for a variety of applications, including surface modification, thin-film coating, sterilization, treatment of gases, aerodynamic flow control, and energy-efficient lighting devices [1-3]. Depending on the gas, electrical parameters, and electrode configuration, these discharges can operate in either the classical filamentary mode or in a diffuse mode [4-5]. However, the filamentary mode can be too restrictive for some applications, such as surface coating. On the other hand, the conditions required to obtain a diffuse DBD can also be restrictive.

Diffuse DBDs at atmospheric pressure have been obtained in noble gases like helium, argon, neon, and nitrogen with a Penning admixture [5]. In nitrogen, the ionization level is too low to allow the formation of a cathode fall. Consequently, the electrical field is quasi-uniform over the discharge gap, similar to low-pressure Townsend discharges, and the discharge that is obtained is called Atmospheric Pressure Townsend Discharge (APTD) [5]. Compared to glow discharges obtained in noble gases, the conditions required to obtain an APTD are more restrictive. For example, the APTD can be obtained only at low frequency (from a few Hz to a few kHz, depending on the experimental conditions), and a production of seed electrons is necessary when the electric field is low to obtain the Townsend breakdown.

Several studies have been conducted since 2000 to explore the use of diffuse discharges at atmospheric pressure for plasma coating [6-7]. This type of discharge can produce a dense and uniform coating at atmospheric pressure with a simple, robust, and low-cost configuration [6]. However, there are limitations such as the concentration of precursor, the dissipated power, and the type of substrate. The diffuse discharge is prone to perturbations, which can cause a transition to the filamentary discharge. This transition can affect the discharge mechanism and the resulting coating (Figure 1).

This work aims to synthesize and discuss the technical and scientific challenges for depositing thin films using a diffuse discharge at atmospheric pressure. This work aims to review and discuss the technical and scientific challenges faced when using a diffuse discharge at atmospheric pressure to deposit a uniform thin film. Solutions to improve discharge uniformity, power dissipation, reduce sensitivity to precursor concentration, and increase deposition rate mainly in the case of discharges obtained in N_2 will be brought to the fore.

Keywords: DBD, APTD, electrical discharge, PECVD, coating, thin film, process, materials.

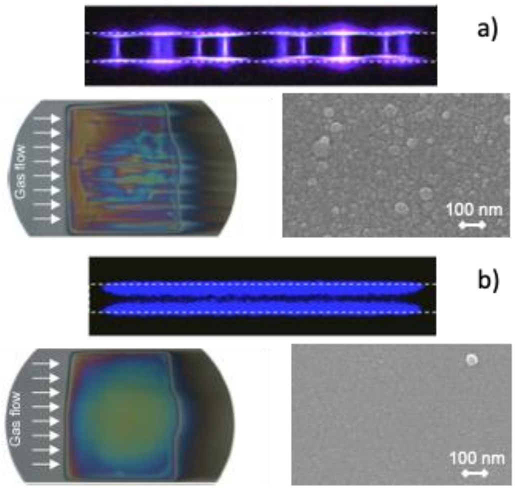


Figure 1: Comparison of a coating realised with: a) a filamentary discharge and b) a diffuse discharge in N_2 with N_2O and HMDSO for the same discharge power (10 W/cm^3)

References:

1. S. Samukawa *et al.*, J. Phys. D: Appl. Phys. **45** (2012) 253001
2. I Adamovich *et al.*, J. Phys. D: Appl. Phys. **55** (2022) 373001
3. U. Kogelschatz, Plasma Chem Plasma P **23** (1), 1-46 (2003)
4. R. Brandenburg, Plasma Sources Sci. Technol. (2017) 26 053001
5. F. Massines *et al.*, Eur. Phys. J. Appl. Phys. 47, 22805 (2009)
6. F. Massines *et al.*, Plasma Process. Polym. 2012, 9, 1041–1073
7. A Fridman *et al.*, 2005 J. Phys. D: Appl. Phys. 38 R1

Plasma-Assisted Low-Temperature Curing of Polysilazane Coating on Aluminum Substrate

Pedram Ghourchi Beigi, Richard Krumpolec, Leila Zahedi, Monika Stupavská, Dušan Kováčik

Department of Plasma Physics and Technology, CEPLANT – R&D Center for Plasma and Nanotechnology Surface Modifications, Faculty of Science, Masaryk University, Brno, Czech Republic

Abstract:

Traditional coatings, primarily organic coatings, have been used for centuries for surface protective and optical purposes; however, their barrier effects may be compromised by the penetration of reactive ions, as well as oxygen and water, followed by some defects on the protected surface and affect its properties. Over the past two centuries, substantial attention has been paid to the research on silicon-based polymers as a proper material for coating due to their liquid nature, which makes them suitable for various cost-effective coating techniques and their thermal and chemical stability. [1]

Polysilozanes (PSZ), known by their Si-N-Si polymeric backbone, have attracted significant interest among these coating materials. The moisture sensitivity of the Si-N bond presented in the backbone, which yields them as an exciting material for the coating application, can adhere firmly to the hydroxyl group presented at the surface of various kinds of substrates, e.g., glass, metals, etc. This coating was developed based on specific features, including anti-fouling, anti-corrosion, and more properties. The crosslinking process of PSZ, which means the replacement of nitrogen in the backbone by oxygen, usually happens within a high-temperature process (>150°C). [2] UV radiation, moisture-induced curing [3], and the addition of nucleophilic catalysts [4] are alternative ways for this process, but they have their drawbacks.

In this work, the PSZ solution is deposited on the aluminum (Al) substrate using spin-coating. Prior to deposition, the Al substrates are functionalized by exposing them to the ambient air plasma generated by Diffuse Coplanar Surface Barrier Discharge (DCSBD) for five seconds at the optimal distance of 0.3 mm. The novel method employed for curing involves introducing hydroxyl groups to the coating using a low-temperature atmospheric-pressure plasma device, specifically the Multi-Hollow Surface Dielectric Barrier Discharge (MSDBD), highlighting its superiority over traditional nucleophilic catalyst addition. After the deposition, the curing process was done for 30 seconds at a distance of 5 mm from the electrode of MSDBD while its power was fixed at 25 watts. To understand the crosslinking of the PSZ coating, Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Photoelectron Spectroscopy (XPS) are utilized. The surface properties of the coatings were characterized by water contact angle (WCA) and surface free energy (SFE) measurements, pull-off bonding strength tests, and pencil hardness tests. As a case study, all the samples were analyzed for up to 14 days after curing using the plasma curing method and normal curing under ambient conditions.

Keywords: Polysilazane, Polymer coating, Low-temperature, Atmospheric-pressure plasma, Plasma curing, MSDBD

References:

1. Colombo, P., Mera, G., Riedel, R., Soraru, G. D. (2010). Polymer-derived ceramics: 40 years of research and innovation in advanced ceramics. *Journal of the American Ceramic Society*, 93(7), 1805-1837.
2. Kozuka, H., Fujita, M., Tamoto, S. (2008). Polysilazane as the source of silica: the formation of dense silica coatings at room temperature and the new route to organic–inorganic hybrids. *Journal of sol-gel science and technology*, 48, 148-155.
3. Müller, S., de Hazan, Y., Penner, D. (2016). Effect of temperature, humidity, and amino alkoxysilane additive on the low-temperature curing of polyorganosilazane coatings studied by IR spectroscopy, gravimetric, and evolved gas analysis. *Progress in Organic Coatings*, 97, 133-145.
4. Furtat, P., Lenz-Leite, M., Ionescu, E., Machado, R. A., Motz, G. (2017). Synthesis of fluorine-modified polysilazanes via Si–H bond activation and their application as protective hydrophobic coatings. *Journal of Materials Chemistry A*, 5(48), 25509-25521.

An engineering and a chemical approach to overcome inhibition of polymerization during open air atmospheric pressure plasma deposition of poly(ethylene oxide) coatings

T. Dekoster [1,2,*], R. Vos[2], W. Van Roy[2], B. Nisol[3], K. Jans[2], A. Delabie[2,1]
[1] Kwantumchemie en Fysicochemie, KU Leuven, Leuven, Belgium
[2]Imec, Kapeldreef 75, Leuven, Belgium
[3]Molecular Plasma Group, Gaston Geenslaan 1, Leuven, Belgium

Abstract:

Cold atmospheric pressure plasma jets (CAPjets) are designed to operate in open air, which reduces the equipment cost and allows for a scalable production process. Thanks to these advantages, CAPjets attract attention from a wide variety of industries with applications including adhesion improvement in automotive, barrier coatings for food packaging, hydrophobic coatings in the textile industry and antifouling coatings for biomedical and healtcare applications. Antifouling coatings prevent biofouling, which is an ongoing challenge in the biosensor industry. Biofouling is the undesired binding of proteins and cells to the sensor surface and limits sensor performance and lifetime (1). Atmospheric Pressure Plasma Deposition (APPD) of poly(ethylene oxide) (PEO)-like antifouling coatings provides a scalable, solvent free way to reduce biofouling for reliable biosensor operation. In this work we demonstrate the impact of open-air processing on the composition and properties of PEO-like coatings by APPD with vinylether precursors using argon as a plasma gas. The open-air environment inhibits polymerization as indicated by low deposition rates, oxygen incorporation in the coatings and instability of the coatings in water. First, we demonstrate how we can improve the composition and stability of the coatings by appropriate nozzle design and by deposition in an environment with lower air content. Using fluid dynamics simulations the nozzle design can be optimized such that the air content in the reaction area can be reduced. The resulting PEO-like coatings are stable and antifouling for an antibody solution and inhibit adhesion of human fibroblast cells (Figure 1). Second, we discuss the advantages and disadvantages of precursors with methacrylate groups instead of vinyl groups. There is a trade-off between coating stability in water and antifouling properties. The obtained insights can contribute to the development of atmospheric pressure plasma deposition processes for a wide range of polymer coatings with controlled composition and properties for various applications. Furthermore, it can contribute to the industrialization of open-air plasma deposition processes.

Keywords: Antifouling, Atmospheric pressure plasma, Poly(ethylene glycol), inhibition of polymerization, poly(ethylene oxide) chemical vapor deposition, plasma-enhanced chemical vapor deposition, open-air processing, organic thin films, polymer coatings

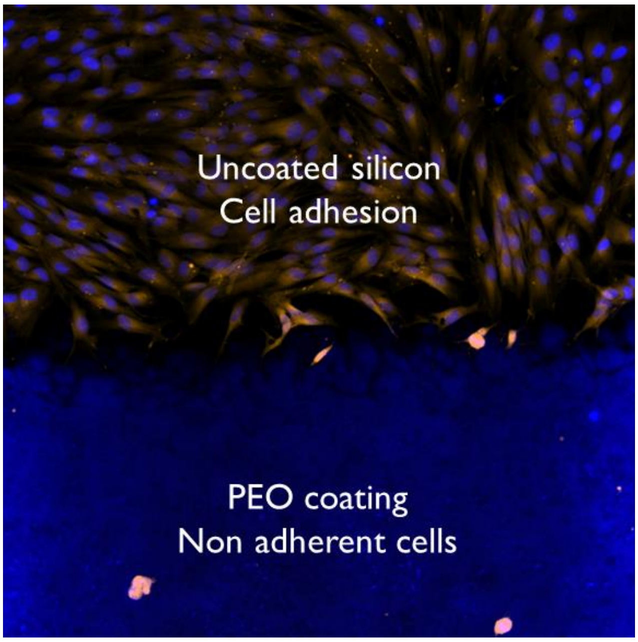


Figure 1: Fluorescence microscopy image demonstrating the antifouling properties of the PEO coating: the upper half is untreated silicon, the lower half is coated with PEO. The fibroblast cells do not adhere to the PEO coating, while they do adhere to the silicon. Color codes: Blue: nucleus stain, Yellow: stain of living cells.

References:

1. A.N. Raditya, D. O'Hare, J. Electrochem. Soc. 167 (2020) 127503.

Study of adhesion promoter coatings, obtained by atmospheric pressure plasma, on granulous substrates

I. Bacquet [1,2], C. Guyon[1], M. Yonger[2], M. Tatoulian[1]
[1] Institut de recherche de ChimieParistech, PSL Research University (UMR 8247)
[2] Saint-Gobain Research Paris, Aubervilliers

Abstract:

Composite materials are high performance materials**. They involve interfaces between organic and non-organic phases, on which the mechanical properties and durability of the material depend.** Material failures often occur at the reinforced/matrix interface. To overcome this problem, a number of chemical and physical surface treatments for assemblies have been developed in recent years. Because of the disadvantages related to wet surface treatments (toxicity, regulations...), and when possible, in industrial processes**, dry surface treatments** are preferred to modify the surface of materials (glass, polymers...). It is therefore important to evaluate a substitution of existing liquid processes, considering the changes in REACH regulations known to date and integrating perfectly into a continuous industrial process.

Plasma treatment is therefore an interesting solution because it offers a more stable and durable surface modification than any other method. This technique, which can be adapted to **substrates with complex shapes**, allows a **wide variety of surface modifications**: etching, grafting of reactive functions on a few Å (functionalization). Plasma can also ensure the deposition of inorganic/organic layers of controlled chemistry and structure over a thickness of a few nm, by radical polymerization under plasma (PE-CVD). However, conventional plasma treatment has several disadvantages such as vacuum (interruption of assembly lines for several hours) and limitation of the size of the treated parts. One of the solutions currently being developed to overcome these disadvantages is the use of **plasma at atmospheric pressure**.

Therefore, particles were treated with an atmospheric pressure plasma jet. Those 3D substrates were functionalized with amino groups[1,2] to improve adhesion with resins (organic phases). The plasma parameters were evaluated to optimize the homogeneity of the treatments and the efficiency of the coating for adhesion improvement. The characterization methods used were acid orange titration for amines quantification. X-Ray Photoelectron Spectroscopy analysis, Optical Emission Spectroscopy and Mass spectrometry were done to understand the mechanisms involved into the plasma deposition and the composition of coatings. Finally, adhesion tests were completed on grains substrates to evaluate the adhesion improvement obtained thanks to the plasma treatments and revealed promising results.

Keywords: Atmospheric pressure plasma, grains treatments, Adhesion, amines functionalization.

References:

1 Morand, G. *et al.* Investigation of 3-aminopropyltrimethoxysilane for direct deposition of thin films containing primary amine groups by open-air plasma jets. *Plasma Processes and Polymers*, doi:10.1002/ppap.202200047 (2022).

2 Rao, X., Hassan, A. A., Guyon, C., Ognier, S. & Tatoulian, M. Plasma deposited high density amines on surface using (3-aminopropyl)triethoxysilane for assembling particles at near-nano size. *Materials Chemistry and Physics* **240**, doi:10.1016/j.matchemphys.2019.121974 (2020).

Effect of Positive Voltage Pulses in HiPIMS on the Ti-PEEK Interface Formation

L. Cvrček [1,*], V. Nehasil [2], M.Buřil [1]
[1] Department of Materials Engineering, Czech Technical University in Prague, Prague, Czech Republic
[2] Department of Surface and Plasma Science, Charles University, Prague, Czech Republic

Abstract:
The application of metal coatings on polymers is limited in many technologies due to the electrical non-conductivity of the polymers. In the case of magnetron sputtering, it is not possible to simply apply a negative bias to the non-conductive material to enhance the ion bombardment, which can affect the formation of the coating-substrate interface and subsequent coating growth. The new perspective method for coating non-conductive materials is HiPIMS (high-power impulse magnetron sputtering) (Figure 1) with a positive pulse [1]. This method was introduced relatively recently, but the effect of a positive pulse has been known for some time in pulsed direct current (DC) magnetron sputtering [2]. The standard HiPIMS method is based on the application of high power in short (tens µs) negative voltage pulses on magnetron cathodes. For the HiPIMS method with a positive pulse, a negative pulse is followed by a positive pulse that repels and accelerates positive ions from the space in front of the target toward the surface of the growing coating. Then, the ions can be accelerated to polymer materials including both non-conductive and conductive polymers. The coating may, for example, reduce the abrasion of the polymer surface, be used as an electrode on conductive polymers, or change the surface properties of polymer implants. In all of these cases, the surface treatment is ensured by applying a metal coating, which must have a very good adhesion. The aim of the work was to verify the influence of positive pulses on the formation of the adhesion interface between the Ti coating and the polyetheretherketone (PEEK). PEEK is electrically non-conductive, but if it contains carbon fibres (CF-PEEK) or carbon nanotubes (CNT-PEEK), its resistivity approaches the level of conductive materials and the effect of negative bias can be tested on it. Therefore, two variants of PEEK were chosen for the test, a non-conductive one in the basic state and a conductive one filled with carbon nanotubes. The performed analyses were focused on the evaluation of the depth of the implanted Ti ions and, at the same time, on the chemical composition of the Ti coating. The application of a positive pulse on the non-conductive PEEK had the same effect on the depth of the Ti ion implantation as the DC negative bias on the conductive PEEK. The depth of Ti implanted analysed by the XPS method did not exceed 60 nm for any of the variants used. However, a positive pulse had a clear effect on the formation of a chemically pure interface without the formation of an atomic inter-mixed layer.

Keywords: HiPIMS, ion implantation, PEEK, XPS, Ti, interface.

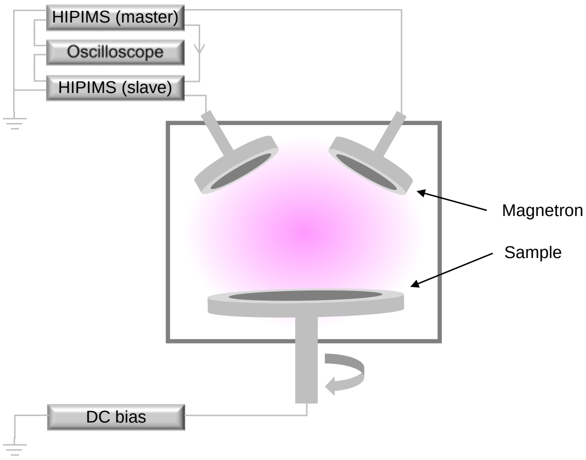


Figure 1: Wiring diagram of the HiPIMS deposition system in configuration with two synchronised magnetrons.

References:

1. H.Du, M.Zanáška, N.Brenning, U.Helmersson, Bipolar HiPIMS: The role of capacitive coupling in achieving ion bombardment during growth of dielectric thin films, Surface and Coatings Technology. vol. 416 (2021).
2. J.Sellers, Asymmetric bipolar pulsed DC: the enabling technology for reactive PVD, Surface and Coatings Technology. vol. 98 (1998) 1245-1250.

Synthesis of carbon micro- and nanostructures by means of DC PACVD

M. Schachinger [1*], F. A. Delfin [1,2], B. Fickl [3], B. C. Bayer [3], C. Forsich [1], D. Heim [1]

[1] Department of Materials Technology, University of Applied Sciences Upper Austria, Wels, Austria
[2] Facultad Regional Concepción del Uruguay, Universidad Tecnológica Nacional, Concepción del Uruguay, Argentina
[3] Institute of Materials Chemistry, Vienna University of Technology, Vienna, Austria

Abstract:

Nanoscale carbon materials, such as graphene, carbon onions and carbon nanotubes, have emerged as critical applications in diverse fields, ranging from simple use as lubricants or corrosion barriers to highly sophisticated roles in energy storage and photocatalysis. In view of the ever-increasing demands on technical components, it is essential to further expand the state-of-the-art methods for the formation of these materials to set new standards for industrial parts, particularly with regard to energy efficiency. In this work, a simple method for the synthesis of carbon micro- and nanomaterials is presented, eliminating the need of laborious pretreatment steps that are normally required for the deposition of carbon nanostructures, allowing easy scale-up to an industrial size. Simple pretreated steel samples of various grades were subjected to plasma deposition via a commercially available DC PACVD system, utilizing an C_2H_2 and Ar flow at a 2 mbar vacuum pressure. The deposition temperature was varied between 400 and 600°C. In addition, a modified composition of the gas phase using HMDSO and N_2 was studied. Characterization of the obtained materials was carried out via SEM, TEM, and Raman spectroscopy. Their chemical composition was analyzed via EDX and XPS. The results show that, depending on the substrate material and the deposition parameters, vastly different carbon structures in the micro- and nanoscale were successfully formed. SEM images reveal contorted layers, marbles, fine wires, tubes and tube forests (Figure 1), exhibiting a high volume to surface ratio. XPS indicates that the primary hybridization type for carbon bonding is sp^2, pointing towards a graphite-like material behavior, i.e., high conductivity. Raman spectroscopy shows that the obtained structures can be classified as stage 1 carbons according to the three-stage-model of Ferrari and Robertson, exhibiting a low FWHM(G) and an I(D)/I(G) ratio between 0,4 and 0,9. Some spectra show a large 2D peak in combination with a RBM, suggesting the formation of multi- and single-walled carbon nanotubes.

Keywords: plasma deposition techniques, carbon micro- and nanostructures, steel substrates, simple pretreatment

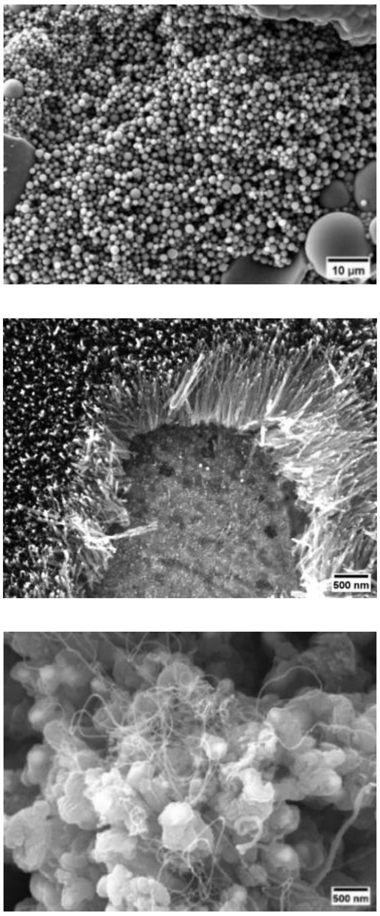


Figure 1: SEM images of different carbon micro- and nanostructures.

References:

1. Ferrari, A.C. (2007) Raman spectroscopy of graphene and graphite: Disorder, electron–phonon coupling, doping and nonadiabatic effects, *Solid State Comm.*, 143, 47-57.

Atmospheric-pressure Plasma Enhanced Chemical Vapor Deposition of size agents on fiberglass

M. Troia [1*], A. Schulz [1], M. Walker [1], M. Haag [2], D. Ben Salem [3], P. Holste [3], A. Knospe [3], C. Dobslaw [4], B. Glocker [4]
[1] IGVP University of Stuttgart, Stuttgart, Germany
[2] ITA RWTH Aachen University, Aachen, Germany
[3] Plasmatreat GmbH, Steinhagen, Germany
[4] PlasmaAir AG, Weil der Stadt, Germany

Abstract:
Fiber reinforced plastics are a class of reinforced materials widely employed in the automotive and aircraft industry [1], to name a few, and consists of a polymeric matrix in which reinforcing glass fibers are embedded. Such fibers provide to the composite material improved mechanical properties such as stiffness, while at the same time preserving its overall light weight. The adhesion at the glass-polymer interface and the protection afforded to it against mechanical damages and chemical corrosion are ensured, in the current state-of-the-art, by a size agent, i.e. an aqueous dispersion, oftentimes silane-based. The main limitations of such approach, which severely limit the performances of the composite material, are caused by the very small fraction of fibers actually coated by such size agent, once it dries up [2]. In the present work, a complement to (or possibly a partial substitute of) the size agent is deposited on the fibers by means of an atmospheric pressure Plasma Enhanced Chemical Vapor Deposition (PECVD) process. The latter is carried out with a commercially available plasma torch by PlasmaTreat, using air and/or nitrogen as feeding gas and several organosilanes as possible precursors, in order to produce a thin film rich of adhesion-promoting functional groups on the fibers surface. Adhesion tests carried out with both preliminar glass plates and single fibers as substrate show promising results for several adhesion-promoting layers, with an increase of pull-off and pull-out force of up to 30% and more than 80%, respectively [3].
As the ultimate goal of the research project is the full integration of the PECVD step in the fibers spinning process at industrial levels, as schematically seen in Figure 1, extensive investigations and optimizations of the in-line industrial module, consisting of a closed housing equipped with several plasma torches, have been carried out beforehand and in parallel with initial testings by means of numerical gas simulations with a Finite Element Method.

Keywords: PECVD, atmospheric plasma torch, adhesion-promoting layer, glass fibers, fiberglass, size agent, industrial applications, numerical simulations, modelling.

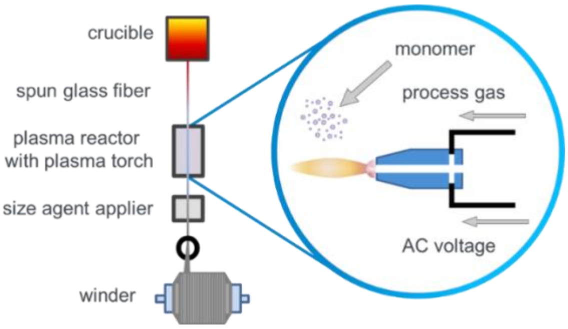


Figure 1: Figure schematically representing of the in-line PECVD process: the extruded spun glass fibers are swept through the plasma reactor, where the adhesion-promoting thin film is deposited in an after-glow process thanks to several atmospheric plasma torches. A size agent ist then applied to th coated fibers, which are finally spun around the winder.

References:

1. Cech, V., Knob, A., Hosein, H. A., Babik, A., Lepcio, P., Ondreas, F., Drzal, L. T. (2014) Enhanced interfacial adhesion of glass fibers by tetravinylsilane plasma modification composites, *Part A: Applied Science and Manufacturing* 58, 84-89.
2. Witten E., Mathes V. (2022) Der europäische Markt für Faserverstärkte Kunststoffe. *Composites*.
3. Haag M., Troia M., Schulz A., Walker M., Delfs P., Dobslaw C., Glocker B. (2022), Plasma coating of glass fibres during the production process for superior adhesion properties in glass fibre reinforced composites, *Proceedings of Aachen Reinforced! Symposium*.
4. Buske C., Delfs P., Haag M., Lüking A., Gries T., (2023) Method and apparatus for manufacture of a fiberglass product, and fiberglass product and use thereof (German Patent DE102021124953A1).

Non-thermal plasma in nanoscale: application potential in the preparation of silicon quantum dots and tailoring of their surface chemistry using plasma-activated liquids.

P Galář [1,*], F. Matějka[1], J. Kopenec[1] and K. Kůsová[1]
[1] Department of Thin Films and Nanostructures, Czech Acadamy of Sciences, Prague, Czech republic

Abstract:
Nanomaterials have become an integral part of modern technologies. To enhance their applicability, researchers continually seek new ways to economically and ecologically optimize their production while preserving their unique properties. One of the most challenging taks in this effort is synthesis of quantum dots (QDs) and nanocrystals (NCs) in general, as their properties depend not only on extremally limited size and structure but also on surface chemistry. Particularly challenging is the production of QDs composed of covalent bonds, such as silicon QDs, which, due to the strength of these bonds, cannot be easily prepared through wet chemistry. Lately, it has been shown that the utilization of non-thermal plasma (NTP) could significantly impact these efforts and can be a way to their commercial synthesis.

Here, we systematically focus on the development of SiQDs synthesis using low-pressure NTP systems. Through adjustments of the synthesis conditions, we can modify not only QDs size but also the surface free energy of the resulting QDs, thus we are able to tune their surface chemistry, hydrogen bondind/dissociation conditions, oxidation dynamics and even the ignition conditions of SiQDs, beyond the scope of previously published results (publication under review). Using the two-phase preparation, we can also create core/shell silicon-based structures[1] or bypass the size limitations of standard NTP synthesis systems within the range from 3 to 20 nm and generate significantly larger SiNCs up to 80 nm.

Another key factor in alteration of SiQDs properties is their surface chemistry. Recently, we demonstrated the potential of NTP in this direction by preparing plasmatically activated water (PAW) with a high concentration of nitrogen species together with no trace of peroxide - HiN:PAW.[2] This activated liquid has proven to be highly effective in nitrogen enrichment of the SiNCs' surface, resulting in quantum yield increasing as well as improved dispersibility in water.[3] We have also started pioneering work in generalization of plasma activation to other liquids, i.e. the NTP activation of different organic liquids (PAOL). Despite the enormous potential of PAOL, their preparation is associated with many obstacles, such as their high volatility and flammability. First and foremost, using a unique reaction chamber, we have successfully created a functional PAOL system and carried out activation of several organic liquids differing in chemical structure or polarity. Subsequently, these activated liquids were effectively bound to the surface of SiQDs while simultaneously modifying their photoluminescent and dispersibility properties in various solvents.

Our efforts demonstrate the significant potential for further development of these techniques, leading to possible straightforward, environmentally friendly, and economically viable approaches for fine-tuning the properties of nanostructures.

Keywords: quantum dots, non-thermal plasma, silicon nanostructures, plasma-activated liquids, tailoring surface chemistry, photoluminescence.

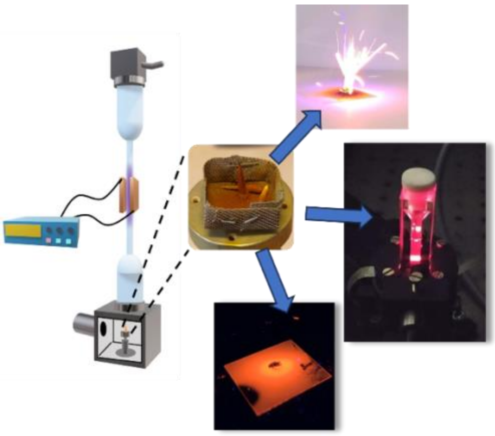


Figure 1: NTP-based SiQDs of various properties (highly flammable, PAW-modified and of high PL generation efficiency).

References:

1. **P. Galář**, J. Stuchlík et al. *Plasm. Process Polym. 2022*, 19, 10.1002/ppap.202100127.
2. F. Matějka, **P. Galář** et al. *Phys. Scr.* 2023, 98 045619 10.1088/1402-4896/acc48e
3. **P. Galář**, J. Khun et al. *Green Chem.*, 2021, 23, 898-911, 10.1039/ D0GC02619K

Adaptation of non-thermal plasma system to overcome size limitations of sythetized silicon nanoparticles

J. Kopenec[1], F. Matějka[1,2], P. Galář[1,2], K. Kůsová[1]
[1] Institute of Physics of the Czech Academy of Sciences, Prague, Czechia
[2]University of Chemistry and Technology, Prague, Czechia

Abstract:

The current hot topic of energy storage technologies centres on the integration of silicon (Si) within anodes of lithium-ion batteries (LIBs). However, a significant challenge arises with the utilization of bulk Si due to substantial volumetric changes during the lithiation process. Silicon nanoparticles (Si-NPs) are one potential way to address this challenge. Presently, there are no problems synthesizing larger Si-NPs exceeding 100 nm in diameter using methods such as milling or thermal pyrolysis. Similarly, methods for synthesizing Si-NPs smaller than 20 nm are well-established and include techniques such as electrochemical etching and non-thermal plasma synthesis. However, a notable gap in synthesis methods exists for Si-NPs ranging from 20 nm to 100 nm, which are the favourable for the anodes due to their good dispersibility, reasonable size and mechanical properties. For these reasons, we focused our research on synthesis of Si-NPs in this size range and their usage in LIBs. We present our outcomes in the synthesis process achieved using an adapted non-thermal plasma system (Figure 1A). To overcome the size limitations, the setup was constructed as a two-stage system. In the first reactor, the sub-10-nm Si-NPs were formed (Figure 1B) and the second reactor was subsequently used to growth of Si-NPs. We also present characterization of size and crystallinity of synthetised Si-NPs. We successfully reached the crystalline Si-NPs with average size around 80 nm (Figure 1C). Furthermore, preliminary results concerning the integration of these Si-NPs into LIBs are also discussed. Specifically, their incorporation into electrodes and subsequent galvanostatic measurements.

Keywords: silicon nanoparticles, nonthermal plasma, synthesis process, lithium-ion batteries, size distribution, crystallinity

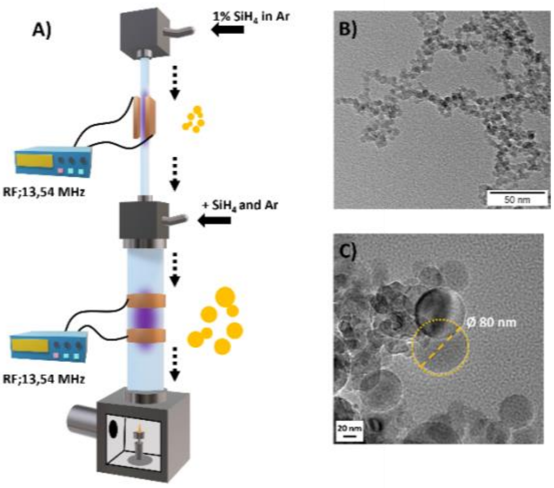


Figure 1: A) Scheme of the adapted non-thermal plasma system. In the first reactor the core Si-NPs was formed from 1% SiH_4 in Ar, the more SiH_4 and Ar was added to the Si-NPs and in the second reactor the enlargement occurred. B) TEM image of sub-10-nm of Si-NPs used as core for enlargement. C) TEM image of the largest synthetized Si-NPs.

References:

1. Kramer, N.J., Anthony, R. J., Mamunuru, M., Aydil, E. S. and Kortshagen, U. R., Plasma-induced crystallization of silicon nanoparticles. Journal of Physics D: Applied Physics, 2014. 47(7): p. 075202.
2. Dworschak, M., Kohlmann, N., **Matějka, F., Galář, P.,** Kienle, L., Schäfer, J., Benedikt, J., Silicon nanocrystal synthesis with the atmospheric plasma source HelixJet. Plasma Processes and Polymers, 2022. 20.

Plasma Jet Sputtering as an Efficient Tool for the Preparation of Transition Metal Oxide Catalysts Supported on Stainless Steel Meshes

P. Topka [1,*], K. Jirátová [1], M. Čada [2], I. Naiko [2], A. Ostapenko [2], J. Balabánová [1], M. Koštejn [1], J. Maixner [3], Z. Hubička [2], F. Kovanda [3]
[1] Institute of Chemical Process Fundamentals, Czech Academy of Sciences, Prague, Czech Republic
[2] Institute of Physics, Czech Academy of Sciences, Prague, Czech Republic
[3] University of Chemistry and Technology, Prague, Czech Republic

Abstract:

Low-temperature plasma sputtering is one of the most important methods used in industry to deposit thin films on various substrates. Using a planar magnetron, thin films of metals or dielectrics can be prepared over large areas using radio frequency source of energy. Recently, an alternative deposition method based on sputtering using a metal nozzle with a flowing gas has attracted attention. Inside the nozzle, an intense discharge is ignited and the working gas (usually argon) flows into a vacuum chamber where the so-called plasma jet enters the plasma [1]. Transition metal oxides are suitable for the abatement of harmful volatile organic compounds by total oxidation [2]. We decided to study a Ni-Co-Mn oxide system from the point of view of the effect of various conditions of plasma jet sputtering on stainless steel meshes and reveal their influence on the properties and activity in the gas-phase total oxidation of ethanol and toluene as model compounds. The Raman spectroscopy proved crystallization and oxidation process during calcination. The XRD showed the formation of NiO-like oxides in the calcined catalysts. The XPS spectroscopy revealed the enrichment of the catalyst surface by Mn. The effect of energy delivery on the performance of the catalysts in ethanol and toluene oxidation was studied. In ethanol oxidation, the specific reaction rate at 200 °C was the highest at mild delivered plasma energy (1.6 kWh) and high magnetron sputtering energy (1.3 kWh). In toluene oxidation, the specific reaction rate at 325 °C was the highest when both plasma jet and magnetron sputtering delivery of energy were high (2.3 and 1.3 kWh, respectively). Thin-film catalysts supported on meshes are promissing due to high catalytic activity at low content of active phase and low pressure drop in the catalytic reactor.

Keywords: low-temperature plasma jet sputtering, transitione metal oxides, stainless steel meshes, volatile organic compounds, total oxidation, structured catalysts.

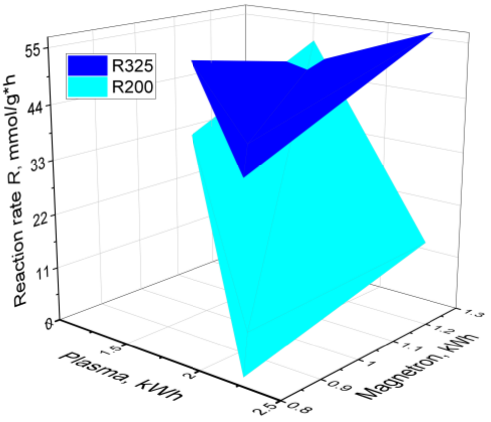


Figure 1: Dependence of reaction rates in the gas-phase oxidation of ethanol (R200) and toluene (R325) on the delivered plasma and magnetron energy.

References:

1. Hubička, Z. (2008), Hollow Cathodes and Plasma Jets for Thin Film Deposition in Low Temperatures plasma, Wiley-VCH, Weinheim, Germany, 2008.
2. Jirátová, K., Babii, T., Balabánová, J., Koštejn, M., Maixner, J., Topka, P., Kovanda, F. (2024), Effect of manganese addition to Ni-Cu oxides on properties and activity in the oxidation of volatile organic compounds, *Catal. Today*, 429, 114463.

General and Scalabe Synthesis of High-entropy Alloy Nanoparticles by Thermal Plasma

Keun Su Kim[1,2*], Martin Couillard[1], Ziqi Tang[2], Homin Shin[1], Daniel Poitras[1], Changjun Cheng[3], Olga Naboka[1], Dean Ruth[1], Mark Plunkett[1], Lixin Chen[3], Liliana Gaburici[1], Thomas Lacelle[1], Michel Nganbe[2], and Yu Zou[3]

[1]National Research Council Canada, Ottawa, ON K1A 0R6, Canada
[2]Department of Mechanical Engineering, University of Ottawa, Ottawa, ON, K1N 6N5, Canada
[3]Department of Materials Science and Engineering, University of Toronto, Toronto, ON M5S 3G8, Canada

Abstract:

High-entropy alloy (HEA) nanoparticles (NPs) comprising five or more principal elements with a near equimolar ratio are emerging materials[1]. Homogenous mixing of large number of elements induces synergy among different elements with strong quantum confinement effects, thus results in unusual combinations of functional properties appealing to broad range of applications such as structural alloys, catalysis, sensing, and energy storage[1]. While general/scalable synthesis of HEA NPs is of particular interest, the controlled incorporation of multiple elements into a tiny particle (< 100 nm) still remains a significant challenge requiring extreme fabrication conditions[1]. Here, we report an ultrafast (< 100 ms), one-step method for the continuous synthesis of HEA NPs directly from a mixture of elemental metals, based on the RF thermal plasma technology[2]. A RF induction thermal plasma jet plasma jet (> 5,000 K) was employed for rapid heating/cooling (10^3–10^5 K/s), and demonstrated the synthesis of HEA NPs (~50 nm) at an unprecedentedly high rate approaching 35 g/h. To demonstrate the generality of our plasma process, we demonstrated HEA NPs of CrMnFeCoNi, MnFeCoNiCu, CrFeCoNiMo and CrFeCoNiCu with different plasma gases of hydrogen and helium. Molybdenum (Mo) and Cu were included specifically because Mo has different physical properties compared with other elements (e.g., low satruration pressure) while Cu is known to be hardly alloyed with other elemental metals due to its positive binary mixing enthalpies. Structural and compositional properties of the newly synthesized HEA NPs were studied thorougly by XRD and HR-TEM analysis, including energy dispersive X-ray (EDX) elemental mapping and EDX line-scanning. The details will be presented. Our work provides a promising route towards scalable synthesis of HEA NPs for their cost-effective applications.

Keywords: high-entropy alloy, nanoparticles, scalable synthesis, thermal plasma

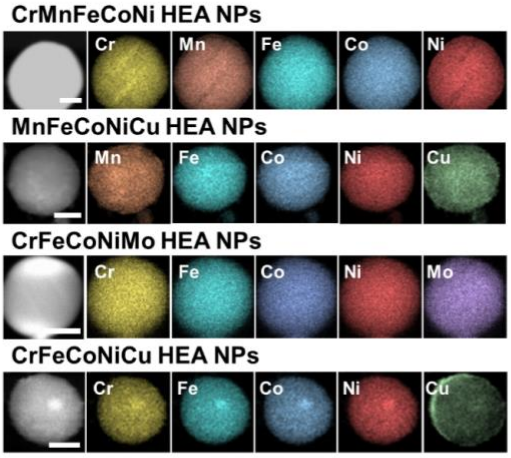


Figure 1: High-angle annular dark-field scanning TEM (HAADF-STEM) images and energy dispersive X-ray (EDX) elemental maps of various HEA NPs synthesized by the RF thermal plasma (CrMnFeCoNi, MnFeCoNiCu, CrFeCoNiMo, and CrFeCoNiCu HEA NPs). Scale bar, 50 nm.

References:

1. Koo, W. T., Millstone, J. E., Weiss, P. S. & Kim, I. D. (2020) The design and science of polyelemental nanoparticles, *ACS Nano*, 14, 6407-6413.
2. Kim, K. S. et al. (2023) (under review)

Synthesis of Simulated Radioactive Post-Detonation Particles by DC Plasma

G. Cota-Sanchez[1], M. Martinez[1], A. Chaudhuri[1], M. Totland[1], J. Garcia-Alonso[1], N. Lee[1]
[1]Canadian Nuclear Laboratories, Chalk River, Ontario, Canada

Abstract:

A radiological dispersal device (RDD) event would purposefully spread radioactive material over an area without the involvement of a thermo-nuclear explosion. This may include a combination of conventional explosives with radioactive material. Post explosion nuclear forensics of an RDD event is conducted through the examination of the resultant radiological and explosive residue and particles. Consequently the methodologies used for forensic analyses are geared towards determining the physical-chemical properties of these residues/particles.

Research activities on post-explosion nuclear forensics of RDD have been undertaken at Canadian Nuclear Laboratories (CNL) [1]. One of the key R&D areas of post-explosion nuclear forencis at CNL involves the use of a direct current (DC) plasma torch to simulate a controlled laboratory scale version of explosion conditions. Produced particles in a laboratory environment can support the analysis in the event of a real RDD event requiring particle analyses.

This work discusses the synthesis of simulated radioactive post-detonation particles by DC plasma. The plasma method is based on the use of a 40-kW DC thermal plasma torch for the decomposition of a powder mix comprised of a combination of radioactive and explosive surrogate materials. Figure 1 shows the experimental set-up used in the tests.

Theoretical work included equilibrium thermodynamic studies performed to estimate the potential compositon of the post-detonation particles produced at the temperatures typical of conventional explosive devices (~4500 K).

Experimental work consisted of several tests performed to evaluate the effect of electrical power, reactor pressure, and plasma gas, on the production of post-detonation particles.

Simulated post-detonation particles were analyzed with a number of characterization techniques, including XRD, SEM and TEM. Both the experimental and theoretical results were compared to determine the plasma conditions that better represent RDD explosions.

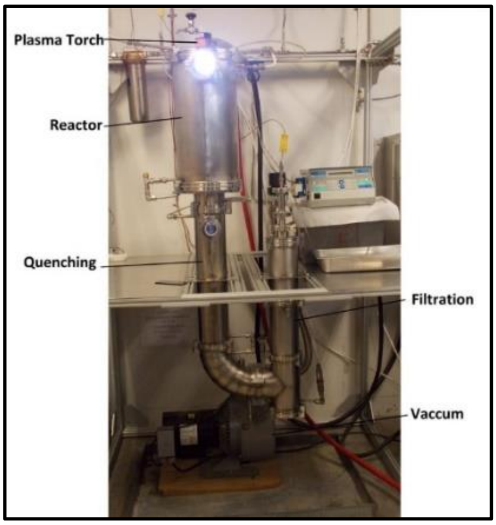


Figure 1: Experimental Plasma System [2]

Keywords: DC thermal plasma, radiological dispersal device (RDD), thermodynamic equilibrium.

References:

1. Chaudhuri, A. et al., (2024). *Particle Production and Characterization for Nuclear Forensics Following a Radiological Dispersal Event*, to be presented at Int. Conf. Nucl. Sec., Vienna, AT, 20-24 May.
2. Cota-Sanchez, G., Turgeon, D. (2019). *Research Activities on Plasma Technology at Canadian Nuclear Laboratories*. 24th 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.

Investigation of ionized metal flux fraction of magnetron sputtering at industrial conditions

P. Vašina[1], J.Hnilica[1], P. Klein[1], S. Debnárová[1], V. Sochora[2], M. Učík[1,3], J. Klusoň[3], M. Jílek[2,3], A. Lümkemann[4]
[1] Department of Plasma Physics and Technology, Masaryk University, Czech Republic
[2] SHM s. r. o., Šumperk, Czech Republic
[3] PLATIT a.s., Šumperk, Czech Republic
[4] PLATIT AG, Selzach, Switzerland

Abstract:

The objective of this study is to present two distinct strategies for enhancing Ionized Metal Flux Fractions (IMFF) in industrial settings, particularly aiming to achieve values close to 40 percent for Titanium (Ti) sputtering. Additionally, we aim to demonstrate the impact of IMFF enhancement on the quality of deposited layers in both non-reactive and reactive processes.

In the first strategy, we conducted experiments in an industrial setting utilizing a deposition system developed by SHM (Sumperk, Czech Republic). This system featured a titanium cylindrical rotating target with a racetrack area of approximately 100 cm². An efficient target cooling system enabled us to deliver up to 35 kW of DC power to the central sputtering cathode, resulting in an exceptionally high ionized metal flux fraction of 23 percent at the substrate level. This value is notably close to what is typically achieved with high-power impulse magnetron sputtering (HiPIMS) technology. The measurement of this high value challenges the commonly accepted notion that IMFF in DC magnetron sputtering is typically only a few percent at the substrate level. This exceptional result is attributed to the equipment's technical advantages, allowing for extremely high-power density. Additionally, we demonstrate the potential to increase IMFF by up to 10 percent using a hybrid deposition system that combines lateral glow discharge with magnetron sputtering. This hybrid technology offers independent control over the ion and neutral fluxes on the substrate, resulting in a pronounced preferential 111 orientation of TiN grains and improved hardness and roughness of TiN coatings compared to standard magnetron sputtering.

The second strategy was implemented in an industrial setting using a deposition system developed by Platit (Platit AG, Salzach, Switzerland and Platit a.s. Sumperk, Czech Republic). Platit introduced a novel cathode, referred to and marketed as the "F-type cathode," which optimally concentrates the total power into a small target area while actively preventing overheating. This approach enables the attainment of high-power density, reaching up to 400 W/cm² in the racetrack region, leading to IMFF values of up to 35 percent. It's important to note that the F-type technology employs standard DC sources but achieves results as HiPIMS. The primary distinction lies in the concentration of power in a small area but continuously delivered for F-type cathode, as opposed to HiPIMS, where power is delivered across the entire target area but in short intervals. Fig. 1 compares IMFF for standard cathode (SCiL) and F-type fordifferent applied powers. Coatings deposited with the F-type cathode exhibit density and smoothness comparable to HiPIMS layers.

In summary, this study explores two innovative strategies for increasing IMFF in industrial settings, shedding light on their potential for enhancing the quality of deposited layers, as was already demonstrated for reactive and non-reactive sputtering of titanium.

Keywords: magnetron sputtering, ionized flux fraction, industry, deposition, PVD, HiPIMS

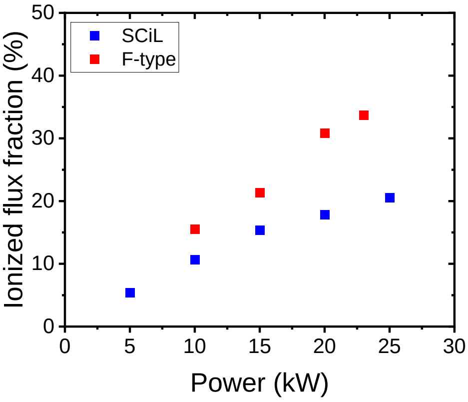


Figure 1: Ionized metal flux fraction as a function of applied power for standard cathode (SCiL) and F-type cathode (F-type).

Active turbulence in a 2D complex plasma with Janus particles

V. Nosenko [1]
[1] Institut für Materialphysik im Weltraum, Deutsches Zentrum für Luft- und Raumfahrt (DLR),
D-51147 Cologne, Germany

Abstract:

Janus particles are polymer microspheres with hemispherical metal coating. When dispersed in plasma, they acquire self-propulsion and act as microswimmers [1,2]. Such dusty plasmas with Janus particles are useful model systems of more complex active matter systems such as living organisms.

A two-dimensional complex plasma containing active Janus particles was studied experimentally. The experiments were carried out in a modified Gaseous Electronics Conference (GEC) rf reference cell. Plasma was produced by a capacitively coupled radio-frequency (rf) discharge in argon at 13.56 MHz. The gas pressure was 0.66 Pa, the discharge rf power was 20 W. A single layer of micrometer-sized plastic microspheres coated on one side with a 40-nm layer of gold was suspended in the plasma sheath above the lower rf electrode.

Due to their self-propulsion, the Janus particles moved around with high velocities greatly exceeding the thermal speed and their suspension was in a highly disordered (gas-like) state. Below, we address the important question of whether this apparently chaotic state has characteristics of active turbulence.

A key feature of turbulence is the energy cascade with inertial interval where a power-law scaling indicates self-similarity of the velocity field. In the classical Kolmogorov theory of turbulence the universal power-law scaling exponent of -5/3 is predicted. In our situation, the energy cascade is quantified by the power spectral density of the particle velocity as a function of the wave number PSD(k). We calculated PSD(k) as the Fourier transform of the Eulerian correlation function $S_2^{\parallel}$ of the Janus particles' velocity, see Fig. 1. Here, the line is a power-law fit with a non-universal scaling exponent of $\approx$-1.2.

The observed power-law scaling of PSD(k) indicates possible presence of active turbulence in the suspension of Janus particles in plasma. Further research is necessary to find out whether it belongs to the same universality class as the inertial turbulence.

Keywords: complex plasma, active matter, Janus particles, plasma coating.

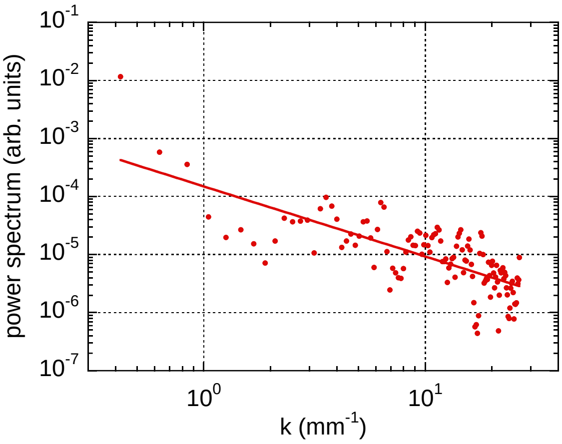


Figure 1: Power spectral density PSD(k) of the Janus particles' velocity. The line is a power-law fit with a non-universal scaling exponent of -1.2.

References:

1. Nosenko, V. (2022), Two-dimensional complex (dusty) plasma with active Janus particles, *Phys. Plasmas*, 29, 123701.
2. 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 sprayed porous Raney Nickel cathodes for alkaline water electrolysis

M. Wetegrove[1], U. Lindemann[1], A. Kruth[1]
[1] Leibniz Institute for Plasma Science and Technology (INP), Greifswald, Germany

Abstract:

The development of a Raney Nickel based electrode for the hydrogen evolution reaction (HER) fabricated by Atmospheric Plasma Spraying (APS) is presented. The goal of the optimization is to achieve a high catalytic activity, as well as a high specific surface area to accommodate the cathodic reaction. The cathode layer's microstructure is characterized using electron microscopy in combination with focused ion beam preparation and EDX. Crystalline phases are identified and characterized by X-ray diffraction. Properties of the layers are correlated with deposition parameters of the atmospheric plasma spray coating process.

Keywords: atmospheric plasma spraying (APS), alkaline water electrolysis, hydrogen evolution reaction, Raney Nickel, catalyst layers.

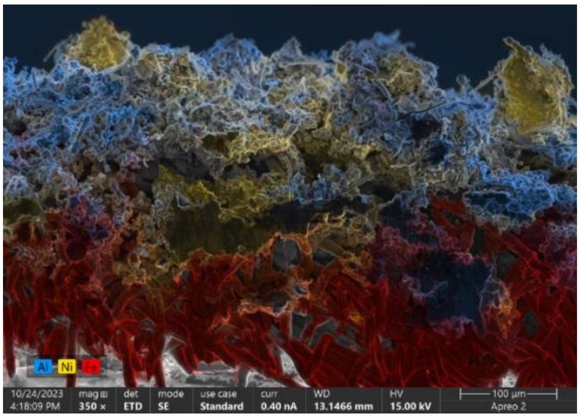


Figure 1: Electron microscopic image showing a plasma sprayed Raney nickel cathode on a stainless steel fleece substrate. Elements are mapped according to their EDX intensities with Al shown in blue, Ni shown in yellow, and Fe shown in red.

Tribology 2024 Session II. D: Coatings and Surfaces Corrosion / Tribological Properties / Physics or Chemistry of Tribo-Surfaces / Nanotribology

Solving tribological challenges in the era of Digital Transformation and supporting Circular Economy as well as Energy and Mobility Transition

E. Badisch [1,*]
[1] AC2T research GmbH, Excellence Centre of Tribology, Wiener Neustadt, Austria

Abstract:

Research in Tribology supports solutions to mitigate the major challenge of our times, the global climate change. As response to this major challenge, Tribology acts as technology enabler to customized solutions in the three thematic foci of scientific and technological relevance. (i) Energy and Mobility Transition, (ii) Circular Economy, (iii) Digital Transformation.

- The Energy and Mobility Transition summarizes the global ambition to replace fossil energy carriers with renewable, CO_2-neutral ones.
- The Circular Economy requires the development of products and processes in a circular manner.
- The Digital Transformation is a process of using digital technologies to enable new value creation business opportunities.

Tribology, which its principal purposes to control friction, minimize wear and optimize lubrication, is a key player in enabling new technologies to achieve climate neutrality. As such a technology enabler, Tribology paves the way for the use of carbon-free energy carriers, including the required components. In the sense of the circular economy, sustainability is the core aspect in the design of new materials and lubricants as well as the replacement of critical raw materials. The rigorous use of data science and methods like machine learning and multivariate statistics in Tribology research will push forward product innovations and efficient processes.

European and national climate goals like the European Green Deal are challenging and pushing the industry towards new sustainable products and processes. This applies in particular to targets relating to clean energy, energy efficiency, the efficient use of natural resources through new technologies and measures of circular economy.

This work presents specific research fields contributing to the above-described thematic foci:

- DLC coatings are applied in many applications to minimize friction; additionally these coatings exhibit extreme wear resistance. In this work, a novel measurement approach is presented which allows the detection of critical operational conditions.
- When using self-lubricating coatings on tools, a controlled friction behaviour up to 600°C can be realized which allows the reduction of conventional lubricants.
- The use of data science methods in Tribology research brings a lot of benefits, e.g. enables early failure detection and prediction of performance and lifetime.

Current research beyond state of the art of operando tribometrology is used to gain insight into the origins of friction and wear. Here, operando techniques for the specific use case in Tribology are developed, e.g.

- dynamic behaviour of tribosystems using vibrometer measurements,
- high-intensity X-ray analysis coupled with an in-situ tribometer, and
- novel set-up of H_2- and NH_3-tribotesting coupled with mass spectrometry.

In conclusion, it can be said that research in Tribology (i) saves energy and resources, (ii) ensures functional reliability and safety, and (iii) enables solutions to achieve climate protection targets.

Keywords: Tribology, friction, wear, lubrication, energy efficiency, DLC, self-lubrication, H_2-tribology, C-free energy carriers, sustainability.

Acknowledgements: This work was funded by the Austrian COMET Program (project K2 InTribology, no. 872176 and has been carried out at the Excellence Centre of Tribology (AC2T research GmbH).

Controlling surface tribological properties reduces the environmental impact of engineering technologies.

Giuseppe Carbone
Department of Mechanics Mathematics and Management, Politecnico di Bari - Italy

Abstract:

The demand of adhesives, sealants, hydro-repellent surfaces and frictional material is constantly growing every year. This increasing demand is associated with the requirements of extremely low environmental impact and much better durability and failure resistance of engineering materials and components. It is worth noting that the 23% (about 119 EJ) of the world's total energy consumption originates from tribological contacts. It is estimated that new surfaces, materials, and lubrication technologies may reduce energy losses due to friction and wear by 40%. However, our ability to manipulate and control surface properties as adhesion, friction and wetting is still marginal. Therefore, a deeper understanding of such phenomena is a key point to mitigate the economic losses and environmental impact. Adhesive/anti-adhesive and frictional materials are indeed employed in a wide range of industrial and technological applications, spanning several length-scales from the macro world - construction, motor vehicles, durables manufacturing - to the micro/nano world - MEMS/NEMS devices, microfluidics, biomedical and biotechnological systems, magnetic storage and recording systems. Depending on the type of application, adhesion, friction, and wettability should be either increased or reduced. Tribological properties can be manipulated by controlling surface roughness or by adding specific micro- and nano- structures at the interface. Multi-scale modelling and theoretical approaches aimed to identify proper geometrical sub-structural interfaces represent the fundamental steps towards the fabrication of truly sustainable engineering devices.

Application of Surface Modification Technology for Improving the Tribological Properties of Additively Manufactured Materials

A. Amanov *
Faculty of Engineering and Natural Sciences, Tampere University, Tampere, Finland

Abstract:

Additive manufacturing (AM) offers an excellent capability for designing complex geometries with topology-optimized and near-net-shaped structures [1]. However, materials manufactured by AM technologies usually exhibit deterioration of mechanical, tribological and fatigue properties due to microstructure-related defects such as internal porosity, residual stress, micro-crack, unmelted powder, anisotropy, etc. [2]. In addition, the surface roughness of AMed materials is relatively high, which affects their mechanical and tribological properties. Hence, a post-AM surface treatment has become an attractive strategy for improving the performance of AM parts. In this study, the application of ultrasonic nanocrystalline surface modification (UNSM) treatment was applied to various materials (metals and ceramics) to improve the tribological properties by reducing the surface roughness and modifying the microstructure. Figure 1 shows the LSM images of the as-SLM manufactured Ti-6AL-4V alloy subjected to UNSM-treatment at room and high temperatures. SLM fabricated samples demonstrated a very rough surface due to the partially unmelted powders and gas-induced pores on the surface. Obviously, the UNSM treatment at both temperatures could impinge against those unmelted powders and expel the pores from the surface, which was flattened at the end with no unmelted powders, pores and even cracks. Figure 2 shows the variation in the coefficient of friction for the untreated and UNSM-treated Ti-6AL-4V alloy. It is clear that the as-SLM and UNSM-25C samples demonstrated a similar frictional behavior, but the UNSM-800C sample extended the running-in period. Essentially, a lower coefficient of friction was dominated by the initial roughness of the samples. In contrast, an increase in hardness of the UNSM-800C sample, which came into first contact with the surface of the counterface ball, had harder asperities that could increase the level of plastic deformation. This study provides a more comprehensive discussion of the relationship between the microstructural evolution by UNSM treatment and the tribological properties of AM materials for various applications.

Keywords: Additive Manufacturing, Surface roughness, Nanostructured surface layer, Tribology, Surface modification technology.

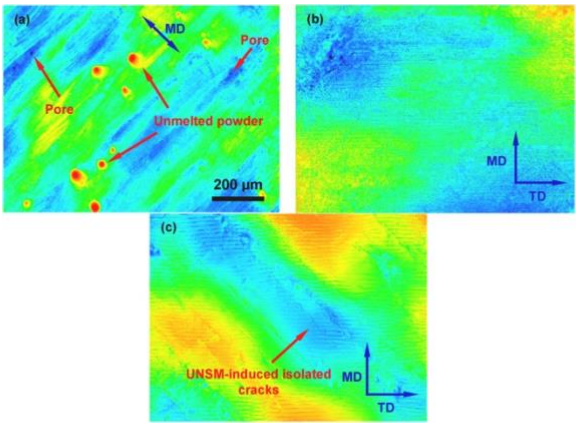


Figure 1: Top surface LSM images of the as-SLM (a), UNSM-25 C (b) and UNSM-800 C (c) samples [2].

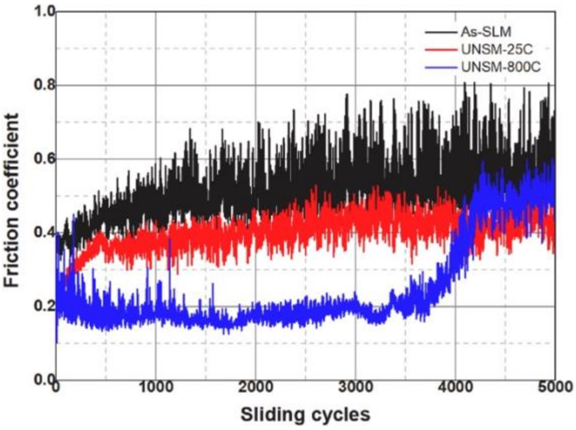


Figure 2: Variation in the coefficient in friction of the as-SLM, UNSM-25 C and UNSM-800 C samples [2].

Reference:

1. Srivatsan, T.S., Sudarshan, T.S., (2016) Additive Manufacturing – Innovations, Advances, and Applications, *CRC Press*, Taylor & Francis Group.
2. Amanov, A. (2021) Advancement of tribological properties of Ti-6Al-4V alloy fabricated by selective laser melting, *Tribology International*, 155, 106806.

Friction-assisted Selective Electrodeposition of FeCoNi Alloy Film on Pre-wear Surface of Bearing Steels

Y. Song, C. Liu, Y. Meng*
State Key Laboratory of Tribology in Advanced Equipment, Tsinghua University, Beijing, China

Abstract:

Remanufacture engineering is important for achieving sustainable development and reduction of carbon emission [1]. Among various repair technologies available for worn parts, alloy electrodeposition is considered to be an efficient surface modification technology to provide coatings which has unique mechanical, chemical and physical properties for aerospace, energy and automotive applications [2, 3]. In electrodeposition process, a monolayer or multilayer coating is formed because of the electrochemical reactions occurred on electrode surface. In previous experimental studies, a series of different coatings were successfully electrodeposited. In this study, the feasibility of friction-assisted electrodeposition on a pre-wear bearing steel surface is demonstrated. Selective electrodeposition of nanocrystalline FeCoNi alloys have been successfully achieved on the localized worn surface zone via the friction-assisted electrodeposition (FAED) method. The surface morphology, microstructure features as well as mechanical performance of the deposited FeCoNi film are quantitatively characterized by using optical proferometer, scanning electron microscopy (SEM) with energy dispersive spectrometry (EDS), transmission electron microscope (TEM) and nano-indentation. The results indicated that the friction load and electrodeposition time has a remarkable effect on the deposited films. The cross-section of the deposited film exhibited uniform element distribution, and the typical amorphous and polycrystalline feature were observed in bottom and middle area of deposited film. Additionally, the as-deposited layer shows a matching mechanical property to GCr15 substrate including hardness, complex modulus and wear resistance. Moreover, the improvement mechanism of friction in electrodeposition process is proposed.

Keywords: remanufacturing engineering, selective electrodeposition, friction assisted deposition, nonaqueous electrolyte, FeCoNi alloys, characterization of the deposited metal films.

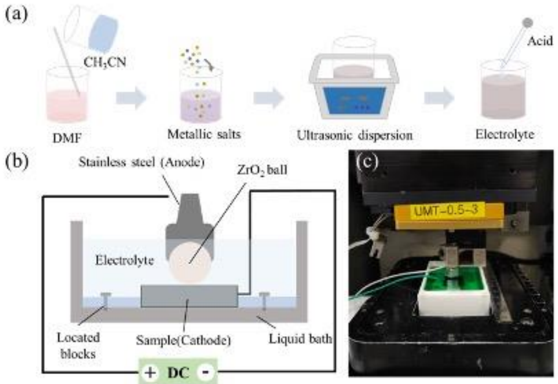


Figure 1: (a) Flow chart of electrolyte preparation; (b) Schematic diagram of friction-assisted electrodeposition; (c) Experimental set-up

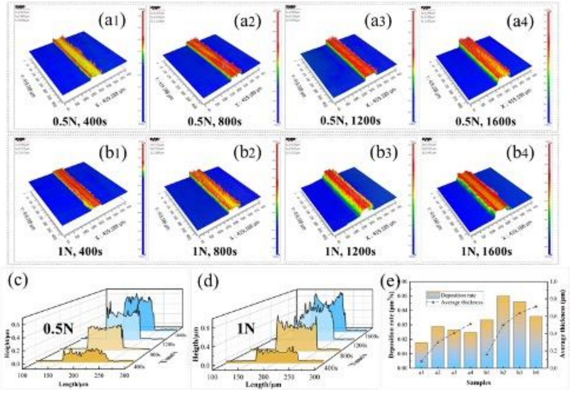


Figure 2: Evolution of 3D surface topography with electrodeposition time under the friction loads of (a1-a4) 0.5N and (b1-b4) 1N and cross-section profile comparison of the deposited film for (c) 0.5N and (d) 1N; (e) deposition rate and average thickness of deposits.

References:

1. Xu, Y., Gao, F., Zhang, B., Nan, F., Xu, B.-S. (2013) Technology of self-repairing and reinforcement of metal worn surface, *Advances in Manufacturing* 1(1) ,102-105.
2. Gurrappa, I., Binder, L. (2016) Electrodeposition of nanostructured coatings and their characterization—A review, *Science and Technology of Advanced Materials* 9(4).
3. Reddy, K.S.K.J., Chokkakula, L.P.P., Dey, S.R. (2023) Strategies to engineer FeCoNiCuZn high entropy alloy composition through aqueous electrochemical deposition, *Electrochimica Acta* 453.

Investigation of the tribological properties and tribo-layers of Graphene in Cu metal matrix composites.

Yuxuan Xu [1], Pingping Yao [1, *], Haibin Zhou [2]
[1] State Key Laboratory of Powder Metallurgy, Central South University, Changsha, China
[2] Hunan Province Key Laboratory of Materials Surface/Interface Science & Technology, Central South University of Forestzhemry & Technology, Changsha, China

Abstract:

Cu metal matrix composites (CMMCs) as friction materials are widely used in friction systems of transportation carriers. Always, stable high friction coefficient and minimized wear loss are expected in order to prolong service life. Tribological properties are determined by the energy transportation and physical properties of the CMMCs and counterparts, typically illustrated as the cracks defect and mechanical deformation of CMMCs surfaces and sub-surfaces. CMMCs reinforced by Graphene obtained via the coated Cu particles and powder metallurgy method exhibits superior tribological performances and form the tribo-layer with stable structure and nanomechanical properties. We report the influence of graphene on the mechanical and tribological properties of CMMCs based on the decreasing 50% wear loss and characterization of tribo-layer of worn sub-surface (Figure 1). The enhancement of the graphene contributes to the physical properties and microstructure of the reinforced tribo-layers, of which the elastic modulus and hardness play a major role on the minimized wear loss. In addition, we will introduce new challenges of the utilization of graphene in CMMCs. This study will also evoke our thoughts about how to utilize this mechanism when facing the changes of counterparts such as C/C-SiC composites or different friction conditions.

Keywords: Cu metal matrix composites; Graphene; wear resistance; tribo-layer; micro-mechanical properties.

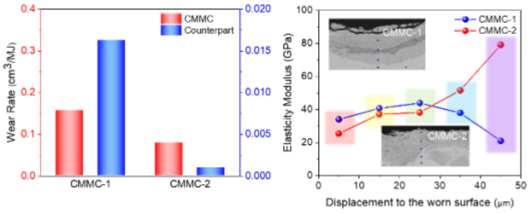


Figure 1: Figure illustrating the approximately 50% decreasing wear loss of CMMC-2 reinforced by graphene as well as the structure and micro-mechanical properties of tribo-layers that we are tempting to investigating: what's the interaction of graphene with Cu matrix to form tribo-layers, and how the energy neutralized and transmitted.

References:

1. H.B. Zhou, P.P. Yao, Y.L. Xiao, K.Y. Fan, Z.Y. Zhang, T.M. Gong, L. Zhao, M.W. Deng, C. Liu, P. Ling. (2019) Friction and wear maps of copper metal matrix composites with different iron volume content, *Tribol Int.*, 132, 199-210.
2. H.B. Zhou, P.P. Yao, Y.L. Xiao, X.B. Liu, K.Y. Fan, T.M. Gong, L. Zhao, M.W. Deng, Z.Y. Zhang. (2023) Influence of ferrochromium type on micro and macro tribology behavior of copper metal matrix composites, *Tribol Int.*, 184, 108409.
3. Z. Y.Zou, C. J. Tu, Y. Xian, L.R. Hong, T.H. Liu, Y. H. Jiang. (2020) Effect of heat treatment temperature on the microstructure of spherical sulfonated graphene particles, *New carbon materials*,. 35, 148-154.

Brake emissions and performance of a WC-coated disc sliding against a NAO friction material

S. Candeo [1,*], A.P. Nogueira[2], G. Straffelini[1]
[1]Department of Industrial Engineering, University of Trento, Via Sommarive 9, Povo, Trento, Italy b
[2]Brembo S.p.A., Stezzano, Bergamo, Italy

Abstract:

Efforts to reduce particulate matter emissions in urban areas have led to the development of strategies targeting various sources, including brake wear in passenger vehicles [1]. The importance of mitigating this source of pollution is underscored by the Euro 7 regulation, which will soon imposes limits specifically to address the brake particle emissions[2].

Conventional tribo-couples usually comprise Non-Asbestos-Organic (NAO) or low-metallic pad materials. The NAO type is recognized for its significantly lower wear, leading to reduced emissions, but the ralteive brake performance under high temperature are very lower compared to low-met materials. Another approach to mitigate brake emissions involves reducing brake disc wear by enhancing its hardness with a hard coating. This coating can be applied using methods such as Direct Energy Deposition (DED) [3] or High Velocity Oxygen Fuel (HVOF) [4] processes. As a matter of fact, , the literature has focused on testing the coated disc with low-met pads. Indeed, the existing literature has mainly focused on testing the coated disc with low-metal pads. The combination of NAO pads with coated discs is reported in only one pin-on-disc study [5], to the best of the authors' knowledge, and no dynamometer studies have been conducted. However, given the low-wear properties of both counterparts, it is crucial to explore their emissions behaviors.

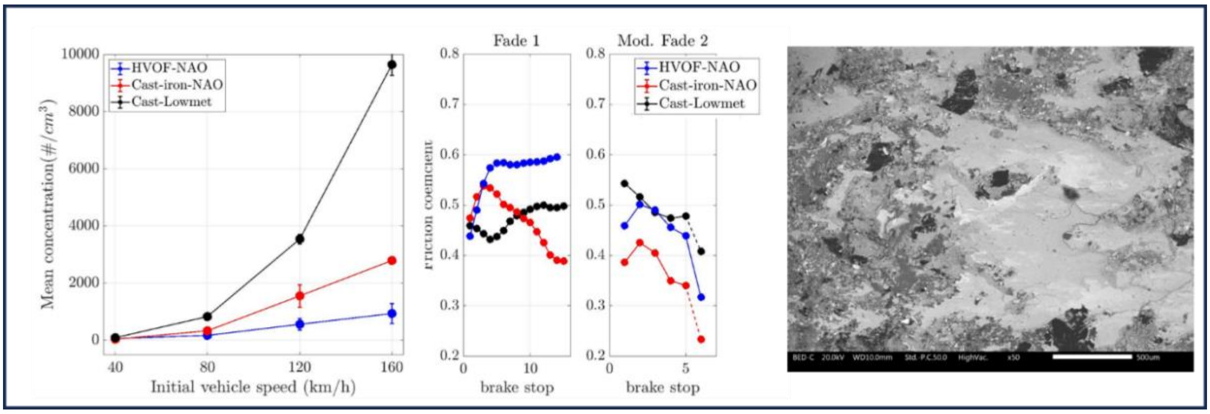
In this study, we investigated the emissions behavior and brake performance of a NAO friction material sliding against a WC-coated disc. The coating was applied using an HVOF technique, as detailed in prior research [6]. Brake emissions were assessed using an Optical Particle Sizer (OPS) and mapped according to the brake cycle outlined in [8] under mild sliding conditions. Interestingly, the NAO-HVOF brake couple exhibited lower emissions compared to the NAO-cast-iron brake couple. Whereas, the brake performance was evaluated using a modified version of the SAE J 2522 AK-Master standard procedure, focusing on the high section. The NAO-HVOF brake couples displayed high sensitivity to pressure and temperature. Additionally, the fade resistance fell between that of the NAO-cast-iron and low-metal-cast-iron brake couples. These observations were supported by SEM-EDXS analysis, revealing high W transfer in the friction layer.

Keywords: brake emissions, brake performance, friction materials, brake cycle, high-temperature testing, fade effect, surface characterization, HVOF.

References:

1. T. Grigoratos and G. Martini, “Brake wear particle emissions: a review,” *Environ. Sci. Pollut. Res.*, vol. 22, no. 4, pp. 2491–2504, 2015, doi: 10.1007/s11356-014-3696-8.
2. “REGULATION OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL on type-approval of motor vehicles and engines and of systems, components and separate technical units intended for such vehicles, with respect to their emissions and battery durability (Euro 7) and.”
3. H. Rajaei, C. Menapace, S. Amirabdollahian, M. Perini, G. Straffelini, and S. Gialanella, “Microstructural and Tribological Evaluation of Brake Disc Refurbishing Using Fe-Based Coating via Directed Energy Deposition,” *Metals (Basel).*, vol. 12, no. 3, 2022, doi: 10.3390/met12030465.
4. [4] M. Federici, C. Menapace, A. Moscatelli, S. Gialanella, and G. Straffelini, “Pin-on-disc study of a friction material dry sliding against HVOF coated discs at room temperature and 300 °C,” *Tribol. Int.*, vol. 115, no. March, pp. 89–99, 2017, doi: 10.1016/j.triboint.2017.05.030.
5. Y. Lyu, M. Leonardi, A. Mancini, J. Wahlström, and U. Olofsson, “Tribology and airborne particle emission of laser-cladded fe-based coatings versus non-asbestos organic and low-metallic brake materials,” *Metals (Basel).*, vol. 11, no. 11, 2021, doi: 10.3390/met11111703.
6. M. Federici, C. Menapace, A. Mancini, G. Straffelini, and S. Gialanella, “Pin-on-disc study of dry sliding behavior of Co-free HVOF-coated disc tested against different friction materials,” *Friction*, vol. 9, no. 5, pp. 1242–1258, 2021, doi: 10.1007/s40544-020-0463-3.

Graphical Abstract


Mean concentration($\#/cm^3$)
10000
8000
6000
4000
2000
0
40
80
120
160
Initial vehicle speed (km/h)
HVOF-NAO
Cast-iron-NAO
Cast-Lowmet
Fade 1
Mod. Fade 2
Friction coefficient
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0
10
5
brake stop
brake stop
HVOF-NAO
Cast-iron-NAO
Cast-Lowmet

Effect of laser texturing on the frictional resistance in reciprocating motion at elevated temperature

S. Wos [1], W. Koszela [1], A. Dzierwa [1], J. Sep [1], P. Pawlus [1,*]
[1] Faculty of Mechanical Engineering and Aeronautics, Department of Manufacturing Processes and Production Engineering, Rzeszow University of Technology, Rzeszow, Poland

Abstract:

Surface texturing leads to an improvement of tribological properties of sliding surfaces by creation of dimples (oil pockets or cavities). These dimples may cause a reduction of friction in fluid, mixed and sliding regimes. Surface texturing can be used also to increases in the seizure and abrasive wear resistances. This technique is very popular [1]. There are many texturing techniques, including burnishing and abrasive jest machining. However, laser texturing is the most popular technique [2].

During co-action between contact elements in reciprocating motion, different working conditions exist in outer and inner zones of stationary elements. Because tribological effects of surface texturing depend on the operating conditions, various dimple patterns were created in the middle part of the steel disc and near the reversal points. Behaviors of variable dimple patterns were compared with those of uniform texturing and untexturing.

The experiments were conducted in lubricated conformal reciprocating motion using Optimol SRV5 tester. Contact region had shape of the ring. Lower disc was the sample and the upper disc was the counter-sample. Both discs were prepared from 42CrMo4 steel of 44±2HRC hardness. Only samples were textured. Before texturing, samples were subjected to grinding. They average roughness height (Ra parameter) of 0.1-0.15 µm.

Surface texturing was performed by laser engraver SpeedMarker 300, made by Trotec®. Laser power was 20 W, focal diameter and length were 64 µm and 254 mm, respectively, pulse duration was 1.5 ns, marking speed was 200 mm/s and pulse repetition rate was 820 kHz.

The stroke of 3 mm and temperature of 80^0 were the constant parameters in tribological tests. Normal loads of 40 and 80 N and frequencies of displacement of 20 and 40 Hz were variable parameters. Investigations were carried out in a stepwise manner, with each subtest duration of 5 min. Each test was repeated 3 times. Before each test, one drop of mineral oil LAN-46 (about 0.08 ± 0.01 ml) was supplied to the contact zone. Kinematic viscosity of this oil at 40°C was 46.0 mm^2/s, kinematic viscosity at 100°C was 6.66 mm^2/s, and viscosity index was 96. This oil was selected, because it contains low number of additives. Before and after wear tests, surface topographies of the lower discs were measured using white light interferometer Talysurfc CCI Lite. Images of the tested disc surfaces were obtained with the Phenom ProX scanning electron microscopy (SEM).

Figure 1 presents contour plots of uniform textured surfaces.

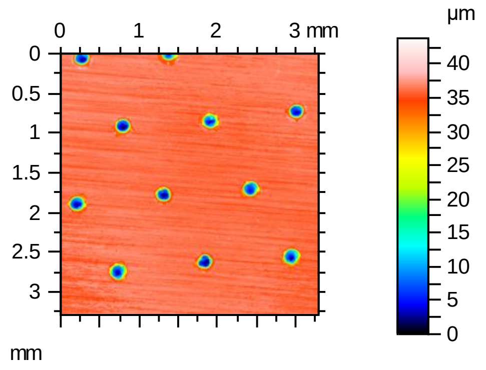


Figure 1. Contour plot of textured surface, pit area ratio was 3%, dimple diameter was 0.2 mm

It was found that the smallest coefficient of friction was achieved for uniformly textured disc surfaces chracterised by pit area ratio od 13% and dimple diamater of 0.4 mm.

Keywords: friction, laser texturing, reciprocating motion

References:

1. Rosenkranz, A., Grützmacher, P.G., Gachot, C., Costa, H.L. (2019) Surface Texturing in Machine Elements − A Critical Discussion for Rolling and Sliding Contacts, *Adv. Eng. Mater.*, 21, 1900194.
2. Etsion, I. (2005) State of the Art in Laser Surface Texturing. *J. Tribol.*, 127, 761-762.

In-depth analysis of the tribological response of PHBV and its blends with PLA via scratch testing

P. M. Martínez-Rubio[1*], M. D. Avilés[1], L. Mínguez[1], R. Pamies[1], and F. J. Carrión-Vilches[1]
[1] Grupo de Ciencia de Materiales e Ingeniería Metalúrgica. Universidad Politécnica de Cartagena.
C/Doctor Fleming sn, 30201 Cartagena (SPAIN)

Abstract:

Biodegradable, bio-based and biocompatible alternatives must constitute the future of polymers, as petroleum limitations, climate change and health priorities are being increasingly prioritized. Poly(3-hydroxybutyrate-co-3-hydroxyvalerate), or PHBV, is a polyhydroxyalkanoate produced via bacterial fermentation, making it promising as a sustanible alternative for food packaging or biomedical applications. However, its thermal and mechanical properties should be optimized in order to replace traditional polymers.

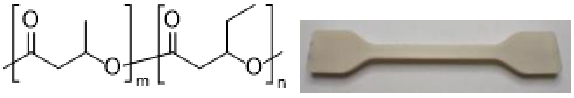


Figure 1: Chemical formula and dumbbell test specimen of PHBV.

As a strategy to overcome these limitations, blending with other sustainable polymers can improve the mechanical behaviour without affecting the sustainability. Poly(lactic acid), or PLA, is a biocompostable polyester obtained from plant startch, that is already well-established in the industry. Its affordability and properties (processability, thermal stability, low crystallinity and relative flexibility) complement those of PHBV, making it an appropriate candidate for blending. In the analysis of the products, tribology serves as a relevant tool to predict the behavior of a material against commonly encountered friction phenomena in the applications described above.

Figure 2: Chemical formula and dumbbell test specimen of PLA.

In this work, dumbbell test specimens of PLA, PHBV and their blends (5 to 25 wt.% of PHBV) were processed via injection molding. Constant load scratch tests (15 scratches, 5 mm long, 5 mm/min) were performed on the samples, evaluating the effect of PHBV content, applied load (5 to 20 N) and direction (longitudinal and transversal to flow direction during processing). Additional progressive scratch tests were performed on PHBV to evaluate the wear mechanism presented by the material at different loads. The evolution of the wear mechanism along the test can be seen in Figure 3. Wear tracks were evaluated using laser profilometry and scanning electron microscopy. All quantitative results were submitted to statistical analysis to assess the effect of each parameter.

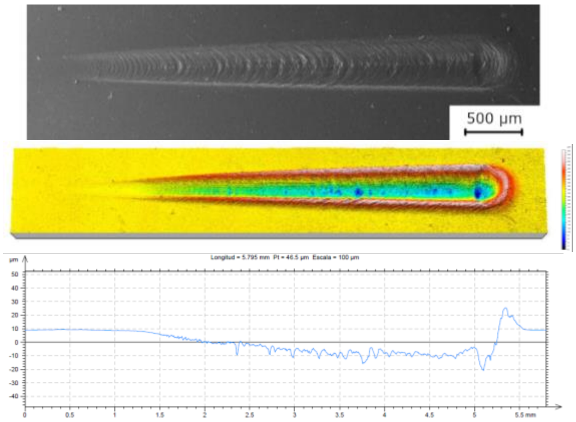


Figure 3: Scanning electron microscopy and laser profilometry of the wear groove after a progressive scratch test on PHBV.

Keywords: biopolymers, tribology, scratch testing, laser profilometry, scanning electron microscopy.

References:

1. Rivera-Briso, A. L., & Serrano-Aroca, Á. (2018). Poly (3-Hydroxybutyrate-co-3-Hydroxyvalerate): Enhancement strategies for advanced applications. *Polymers*, 10(7), 732.
2. Minguez-Enkovaara, L. F., Carrión-Vilches, F. J., Avilés, M. D., & Bermúdez, M. D. (2023). Effect of graphene oxide and ionic liquid on the sliding wear and abrasion resistance of injection molded PMMA nanocomposites. *EXPRESS Polym. Lett.*, 17(3), 237-251.

The Influence of Retained Austenite and Microstructure on the Abrasive Wear Resistance of Bainitic Steels

C.S. Brown [1, 2,*], J.G. Speer [1, 2], E. De Moor [1, 2]
[1] Department of Metallurgical and Materials Engineering, Colorado School of Mines, Golden, CO, USA
[2] Advanced Steel Processing and Products Research Center, Colorado School of Mines, Golden, CO, USA

Abstract:

Bainitic steels have gained recent attention for their promising mechanical properties, including high strength and toughness. This study aims to understand the abrasion resistance of bainitic steels, specifically investigating the influence of retained austenite and variations in microstructure in 9260 and 6150 alloys by employing dry sand/rubber wheel wear testing. Two distinct carbide-free bainitic microstructures were designed using the 9260 alloy, deliberately introducing significant variations in both retained austenite content and morphology. These microstructures were then compared to lower bainitic conditions derived from the 6150 alloy, characterized by comparable hardness. Furthermore, the investigation included and assessment of quench and tempered microstructures from each alloy, providing a comprehensive comparison with the bainitic conditions. Abrasion resistance for each respective microstructure was quantified by monitoring material loss following DSRW wear testing. In tandem with these wear data, scanning electron microscopy was used to visually inspect the wear surfaces. Retained austenite content and sample hardness were additionally obtained for each sample both before and after wear testing to provide support to trends seen in wear data. With hardness held constant, this study aids in the understanding of the complex relationships between microstructure and abrasion resistance of industrially relevant, high-strength steel alloys.

Keywords: steel, abrasion, retained austenite, bainite, dry sand/rubber wheel, microscopy

Microstructural evolution of scuffing in self-mated steels measured in-situ during sliding

S. Berkebile [1,*], C. Lorenzo Martin [2], D. Bachnacki [1,2], F. Ahmed Koly [3], N. Murthy [1], S. Walck [1], J.-S. Park [2], P. Kenesei [2], D. Burris [3], O. Ajayi [2], S. Liu [4], A. Butler-Christodoulou [2], A. Bhattacharjee [3], Q. J. Wang [4], B. Gould [2]
[1] DEVCOM Army Research Laboratory, Aberdeen Proving Ground, MD 21005, USA
[2] Argonne National Laboratory, 9700 S. Cass, Avenue Lemont, IL 60439, USA
[3] University of Delaware, 130 Academy St., Newark, DE 19716, USA
[4] Northwestern University, 2145 Sheridan Rd., Evanston, IL 60208, USA

Abstract:

A type of severe wear called “scuffing” occurs when materials in sliding mechanical contacts undergo significant adhesive and plastic deformation that propagates quickly through the contact area. As with other tribological processes, the active and non-equilibrium processes involved are difficult to measure due to experimental inaccessibility of the moving interface. Therefore, our understanding of material processes during scuffing is limited and data is lacking for comparison to models and simulations. We will present real-time characterization of steel crystallographic microstructure during scuffing failure of lubricated steel on steel in reciprocating mechanical contacts. High-energy synchrotron radiation was employed to characterize the steel microstructure at the interface during active sliding with micrometer resolution via X-ray diffraction in transmission geometry (Figure 1). A larger volume of the steel from the interface to the bulk was also mapped ex situ in the same setup before and after active sliding. Focused ion beam microscopy was used to observe the resulting microstructural distribution and shape of crystallites for comparison to the crystallographic diffraction results. Changes in crystal lattice parameter revealed inhomogeneous strain occured during scuffing that was compressive in the sliding direction and tensile in the loading direction (Figure 2). X-ray diffraction peak width increases accompanied grain refinement in the first few micrometers at the surface. The experimental results correlated well to simulations of strain conducted at different coefficients of friction with asperity contacts.

Keywords: wear, scuffing, steel, microstructure, X-ray diffraction, stress/strain, in situ observation.

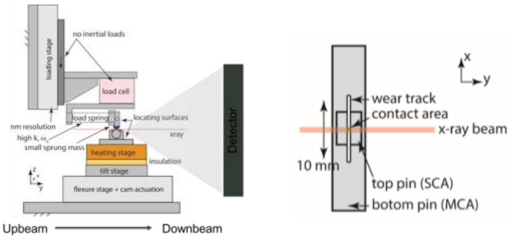


Figure 1: Schematic of tribometer with respect to X-ray beam and detector from side view (left). Schematic of X-ray beam passing through crossed steel cylindrical pins from top view (right). Top pin is 0.5 mm thick, bottom pin reciprocates 10 mm against stationary top pin.

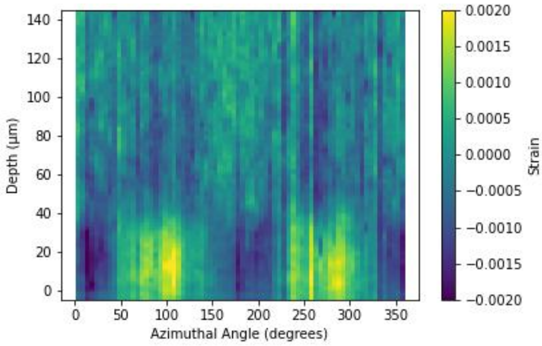


Figure 2: Strain with depth from surface and azimuthal scattering angle in AISI 52100 steel demonstrating compressive strain in the sliding direction and tensile strain in the loading direction to a depth of 40 micrometers from the surface after scuffing has occured. Azimuthal angles 0° and 180° correspond to the sliding direction; 90° and 270° to the loading direction.

References:

1. Ajayi, O. O., et al. (2011). Scuffing mechanism of near-surface material during lubricated severe sliding contact. *Wear*, 271(9-10), 1750-1753.
2. Yagi, K., et al. (2016). Simultaneous synchrotron X-ray diffraction, near-infrared, and visible in situ observation of scuffing process of steel in sliding contact. *Tribology Letters*, 61, 1-16.

Measuring Bearing Loads – Practical Implementation of Impedance Measurement in a Machine Tool Spindle

S. Puchtler [1], M. Fett [1], G. Martin [2] E. Kirchner [1]
[1] Institute for Product Development and Machine Elements, Technical University of Darmstadt,
Otto-Berndt-Str. 2, 64287 Darmstadt Germany
[2] HCP Sense GmbH, Robert-Bosch-Str. 7, 64293 Darmstadt

Abstract:

Tribological contacts are at the heart of manifold mechanical processes. With Industry 4.0, the interest in information about processes parameters increases consistently. Therefore, reasearchers strive to gain valuable information from as close to the process as possible. A promising approach to measure in-situ right in the tribological contact is electrical impedance. In the area of ball bearings, models have been developed based on test rig results [1]. In this work, findings were transferred to the practical implementation in a machine tool spindle for the first time.

First, preliminary tests were carried out on a rolling bearing test bench, cf. Figure 1. On this test rig, interference factors and interactions could be minimized and operating parameters such as speed, axial and radial loads could be reproducibly set and varied. Based on these findings, tests were carried out on an electrically adapted sensor spindle design by Christ Industries. Although the step from the individual bearing to the sensor spindle has greatly increased the system complexity and thus the potential for interference, measured values showed good quality. Subsequently, the sensor spindle was installed and integrated into the whole system of a machine tool by Datron. A force measuring platform was used to measure the forces that occur during the chipping processes. By comparing these forces with the measured impedance, it was possible to confirm the usability of the bearings as sensors. Axial loads, as they occur during drilling processes, are clearly recognizable in the impedance signal. It was possible to derive an almost linear relationship between the axial load and the bearing impedance. Taking the influences of temperature and speed into account, an evaluation model was developed, that allows the interpretation of the measured impedance signal and the derivation of the axial forces acting on the spindle.

The results were achieved as part of a the project “ImpSpin”, funded by the state of Hesse (Germany) and the Distr@l programme.

Keywords: ball bearings, machine tool spindle, in-situ measurement, bearing impedance, condition monitoring, EHL-contact, lubrication film thickness.

References:

1. Puchtler, S., van der Kuip, J., Kirchner, E. (2023) Towards White-Box Modeling of Sensory-Utilized Rolling Bearings, *10th ECCOMAS Thematic Conference on Smart Structures and Materials,* 1635-1645.

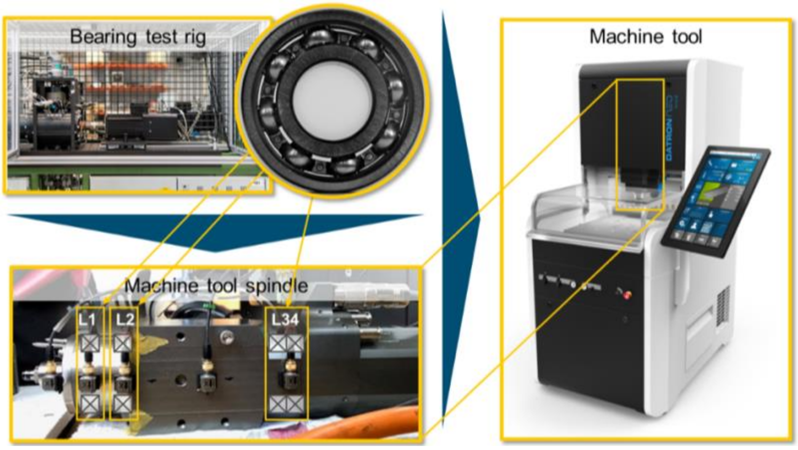


Figure 1: Illustration of the experimental test series. Tests in the bearing test rig were followed by free running tests of the machine tool spindle and chipping test in a machine tool.

$BaTiO_3$ /UHMWPE Composites for Enhanced Performance in Load-Bearing Biomedical Implants

D. Havaldar [1*], Z. Starý[2], L. Cvrček[1], J. Walter[1], Z. Jeníková[1], K. Pawar[3]
[1] Department of Materials Engineering, Czech Technical University in Prague, Prague, Czech Republic
[2] Department of Polymer Processing, Institute of Macromolecular Chemistry, Czech Academy of Sciences, Prague, Czech Republic
[3] School of Nanoscience and Biotechnology, Shivaji University, Kolhapur, India

Abstract:
The pursuit of advanced biomaterials for load-bearing applications in biomedical implants has incited the development of polymer composites tailored for optimal mechanical properties, wear resistance, and biocompatibility. This research emphases on the development and characterization of polymer composites based on Ultra-High Molecular Weight Polyethylene (UHMWPE) reinforced with Barium Titanate ($BaTiO_3$) nanoparticles. In order to assess the suitability of the composite for load-bearing biomedical implants, five distinct combinations of UHMWPE and $BaTiO_3$ were fabricated by with compression molding process (0, 2.5, 5, 7.5, and 10% wt.) and tested with mechanical, surface, and tribological studies.

The mechanical behaviour of the UHMWPE-$BaTiO_3$ composites indicates enhancements in tensile strength, flexural strength, and impact resistance, crucial for ensuring the mechanical integrity required in load-bearing applications. The surface wettability of the composites was analysed with the Contact angle measurements, provided insights into their interaction with biological fluids. Hardness testing was employed to assess the materials' resistance to deformation and wear. Tribological testing, using a pin-on-disc tribometer, explored the friction and wear behaviour, essential for predicting their performance within the human body under load-bearing conditions. The composites were characterized by light microscopy, X-ray Diffraction (XRD), Atomic Force Microscopy (AFM) and Differential Scanning Calorimetry (DSC).

The study reveals the potential of UHMWPE-$BaTiO_3$ composites as biomedical implant materials due to their mechanical strength, hardness, and tribological properties. These composites are suitable for structural integrity and reduced wear applications. Their biocompatibility and therefore applicability for biomedical applications must be further evaluated.

Keywords: UHMWPE, $BaTiO_3$, polymer composites, biomedical implants, load-bearing, mechanical properties, biocompatibility, tribological performance

References:

1. Cruz, M.B., Silva, N., Marques, J.F., Mata, A., Silva, F.S., Caramês, J. (2022) Biomimetic Implant Surfaces and Their Role in Biological Integration—A Concise Review, *Biomimetics*, 7, 74.
2. Al-Shalawi, F.D., Mohamed Ariff, A.H., Jung, D.-W., Mohd Ariffin, M.K.A., Seng Kim, C.L., Brabazon, D., Al-Osaimi, M.O. (2023) Biomaterials as Implants in the Orthopedic Field for Regenerative Medicine: Metal versus Synthetic Polymers, *Polymers*, 15, 2601.
3. Boey, J.Y., Lee, C.K., Tay, G.S. (2022) Factors Affecting Mechanical Properties of Reinforced Bioplastics: A Review. *Polymers*, *14*, 3737.

Role of terminations on the nano-tribological properties of Ti-based MXene layers studied by DFT simulations

E. Marquis [1,*], F.Benini [1], A.Rosenkranz [2], M.C. Righi [1]
[1] Department of Physics and Astronomy, University of Bologna.
[2] Department for Chemical Engineering, Biotechnology and Materials, University of Chile, Chile.

Abstract:
MXenes are a large family of 2D layered transition metal carbides and nitrides passivated by surface terminations. They are commonly denoted by the chemical formula $M_{n+1}X_nT_x$, where M is an early transition metal, X is carbon or nitrogen and T denotes the termination types, which usually consist of -F, -O, -OH in different ratios. These tunable compositional parameters, along with the variable sheet thickness (n), allow for a selective tuning of MXene properties according to the target application [1]. The cability to effectively reduce friction of weakly-interacting layered 2D materials, such as graphene or molybdenum disulfide, has made MXenes appealing for all sorts of tribology applications. Promising performances have been observed in the field of solid lubrication, thanks to the formation of a beneficial tribofilm able to reduce wear and friction [2]. In this regard, computer simulations based on *ab initio* methods represent a useful tool to complement the experimental observations, particularly at the nanoscale.

Here, we employ Density Functional Theory (DFT) to accurately investigate many nano-tribological properties for a large number of Ti-based MXenes ($Ti_{n+1}X_nT_x$). In particular, interlayer adhesion and resistance to sliding are studied as a function of the carbon/nitrogen content (X = N,C), layer-thickness (n = 1,3) and termination type (T = F,O,OH). Binding energies to ferrous substrates (Fe, Fe_2O_3) are also presented, since the layer-substrate adhesion influences the resistance against peeling-off during rubbing.

The accuracy of the computational setup is tested to reproduce the numerical results achieved with higher level of theory. In particular, van der Waals interactions which govern the adhesion between 2D materials are described using the Grimme D2 method with an *ad hoc* choice of the parameters.

We find out that MXene interlayer adhesion is notably affected by the surface termination, while the layer thickness and carbon/nitrogen content play a minor role. The analysis of the potential energy surface (PES) corrugation, performed considering different external applied loads, confirms that MXene' interfacial properties are governed almost exclusively by the passivating groups. A comparative analysis revealed that -O and -F terminations can induce interlayer slipperiness comparable to that of well-known solid lubricants, such as graphene and MoS_2. The binding energy of MXenes on iron and iron-oxide is also governed by the termination type. Our results suggest that the control of surface chemistry during the synthesis of MXenes should be subject of intense research, since surface terminations strongly affect their resulting tribological behaviour (at least at the nanoscale).

Keywords: **2D materials; MXenes; nanotribology; Density Functional Th eory (DFT)**

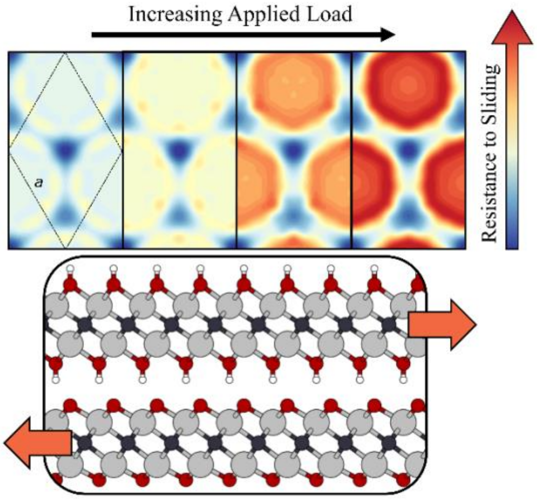


Figure 1: Schematic showing the Potential Energy Surface (PES) corrugation as a function of the applied load for an interface composed by two homogeneously terminated MXene layers $Ti_2C(OH)_2@Ti_2CO_2$.

References:

1. Gogotsi Y., Anasori B. (2019) The rise of MXenes, *ACS Nano*, 13, 8491–8494.
2. Chhattal M., Rosenkranz A., Zaki S., Ren K., Ghaffar A., Gong Z., Grützmacher P. G. (2023) Unveiling the tribological potential of MXenes - current understanding and future perspectives, *Advances in Colloid and Interface Science*, 321, 103021.

The Influence of Pin Inclination on Frictional Behavior in Pin-on-Disc Sliding and Its Implications for Test Reliability

Yue, Hongzhi [1,*],; Schneider, Johannes[1]; Gumbsch, Peter[1]
[1] Institute for Applied Materials - Reliability and Microstructure (IAM-ZM) and MicroTribology Center µTC, Karlsruhe Institute of Technology (KIT), Karlsruhe, 76131, Germany.

Abstract:

The pin-on-disc test is a widely employed method for investigating the friction and wear per-formance of materials in conformal contact. In a typical pin-on-disc system, self-aligning pin holders are frequently utilized to ensure proper surface contact. This study reveals that in such a setup, pin inclination has a significant impact on test reliability, particularly under oil-lubricated conditions, which can overweigh the influence of other parameters such as roughness or texture elements. Utilizing in-situ measurements, we captured the dynamic changes in pin inclination during rotational sliding. Our findings indicate that the pin inclination varies with sliding speed, showing a pitch angle difference of approximately 0.01° as the speed decreases from 2 m/s to 0.04 m/s in our test setup. Importantly, a robust correlation was identified between the friction coefficient and pin inclination, which is supported by the numerical investigation. This study underscores concerns regarding the test reliability of pin-on-disc tribometers, prompting a recon-sideration of the assumptions associated with self-aligning pin holders in such experimental con-figurations.

Keywords: pin-on-disc test, pin inclination, mixed or hydrodynamic lubrication, multi-scale modelling.

A novel MTES/polysilazane hybrid polymer for corrosion protection of stainless steel substrate

P. N. Moghaddam[1,*], M. Parchovianský[1], A. Duran[2], E. Merino[2], Y. Castro[2], I. Parchovianská[1], A. Pakseresht[1]
[1] FunGlass, Alexander Dubček University of Trenčín, Študentská 2, 911 50 Trenčín
[2] Instituto de ceramica y Vidrio (CSIC), Campus de Cantoblanco, 28049, Madrid, Spain.

Abstract:

Polysilazanes are well known as an attractive candidate of coating materials due to its great adhesion to most substrates and its great potential to be modified to obtain tailored properties for a variety of applications [1] [2]. In this work, the hybrid coating of methyltriethoxysilane (MTES) and poly(methylvinyl)silazane (Durazane 1800) is synthesized by sol-gel route to improve corrosion resistance and lifetime of stainless steel substrate. The MTES/polysilazane solution was crosslinked by adding tetra-n-butylammonium fluoride (TBAF) and was deposited on stainless steel by dip coating method. The structure and thermal stability of hybrid matrix are investigated by Fourier transform infrared (FTIR) spectroscopy and thermogravimetric analysis (TGA). FTIR studies demonstrated that with incorporation of MTES to polysilazane the intensity of the Si–N–Si peak was maintained, showing the backbone of the polysilazane was preserved after the addition of MTES. Based on Field emission electron microscopy (FESEM) analysis, the synthesized hybrid coatings were crack-free, smooth, and homogenous. The corrosion resistance of MTES/polysilazane and polysilazane coatings are studied by electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization techniques. The electrochemical results confirmed that the synthesized hybrid coatings improved the corrosion resistance of the stainless steel substrate in 3.5 wt% NaCl solution.

Keywords: Polysilazane, corrosion, stainless steel, thin films

Acknowledgments:

This paper was created in the frame of the project Centre for Functional and Surface Functionalised Glass (CEGLASS), ITMS code 313011R453, operational program Research and innovation, co-funded from European Regional Development Fund. This work is a part of dissemination activities of project FunGlass. This project has received funding from the European Union´s Horizon 2020 research and innovation programme under grant agreement No 739566. The authors also gratefully acknowledge the financial support from the project VEGA no. 1/0171/21.

References:

1. Rossi, S., F. Deflorian, and M. Fedel. "Polysilazane-based coatings: corrosion protection and anti-graffiti properties." Surface Engineering 35.4 (2019): 343-350.
2. Fedel, Michele, et al. "Characterization of polyorganosilazane-derived hybrid coatings for the corrosion protection of mild steel in chloride solution." Coatings 9.10 (2019): 680.

Exploring the Relationship Between Surface Treatment Processes and Tensile Strength in Steel Bonds

P. I. Kovács [1,2], B. Körömi [2], Z. Weltsch [3], M. Berczeli [2,*]
[1] Doctoral School of Multidisciplinary Engineering Sciences, Szechenyi Istvan University, Gyor, Hungary
[2] Department of Innovative Vehicles and Materials, GAMF Faculty of Engineering and Computer Science, John von Neumann University, Kecskemet, Hungary
[3] Department of Automotive and Railway Engineering, ZalaZONE Innovation Park, Széchenyi István University, Gyor, Hungary

Abstract:
This research investigates the impact of heat treatment and various surface modifications on the tensile strength of steel adhesive bonds. The findings reveal a significant link between annealing temperatures and tensile strength, with optimal results observed at 90 minutes and 165°C. Surfaces that underwent turning, sandblasting, and plasma treatment (referred to as type C) consistently surpassed other treatment methods in performance. Notably, the combination of turning, sandblasting, and plasma treatment (type C) demonstrated remarkable tensile strength, achieving 69.06 MPa. Durability tests under various loading scenarios highlighted its superior resilience. Analyzing surface characteristics through roughness metrics, wetting behavior, and Scanning Electron Microscope visuals offered deep insights into the morphological changes triggered by the treatments. Investigations into the chemical composition after plasma treatment revealed crucial changes affecting the surface chemistry, which significantly enhanced the tensile strength to 67.63 MPa. Ultimately, this study sheds light on the complex interplay of factors that determine the strength of adhesive joints in steel, identifying the combined treatment of turning, sandblasting, and plasma as a highly effective method. This revelation piques interest in the detailed mechanisms that drive the enhanced tensile strength observed in these adhesive joints.

Our comprehensive research into the effects of various heat treatments and surface processing on the tensile strength of steel adhesive bonds has produced measurable and significant findings. These results, marked by precise data, highlight several key observations:

- A clear decrease in tensile strength is observed as annealing temperatures increase, with the highest strength of 67.63 MPa recorded at 90 minutes and 165°C. Conversely, samples treated for 45 minutes at 190°C show a reduced strength of 56.62 MPa;

- Surfaces treated by turning, sandblasting, and plasma (Type C) consistently demonstrate enhanced tensile strength. A turned surface alone shows the lowest strength at 21.17 MPa, while turning followed by sandblasting improves strength to 56.16 MPa. The combination of turning, sandblasting, and plasma treatment (Type C) achieves the highest strength at 67.63 MPa;

- Durability tests on the Type C surface reveal its robustness under varying loads, with strengths of 49 MPa for 38 minutes and 47 MPa for 65 minutes;

- Analysis of surface roughness indicates the highest roughness on the turned surface (Ra = 3 μm), with sandblasting reducing roughness to 2.2 μm. The roughness slightly decreases to 2.1 μm after the complete Type C treatment;

- Examinations of wetting behavior and surface energy show the Type C surface to exhibit excellent hydrophilicity, evidenced by contact angles of 7.4° with distilled water and 0° with ethylene glycol, along with a surface energy of 86.6 mN/m;

- Scanning electron microscopy (SEM) confirms the smoothing effect of sandblasting, and energy-dispersive X-ray (EDX) analyses highlight the purifying impact of plasma treatment, significantly eliminating nano-scale contaminants;

- The plasma treatment notably changes the ratios of carbon to oxygen (C/O) and iron to oxygen (Fe/O), indicating a shift in surface chemistry. The Type C treated surface, with a C/O ratio of 24.85, correlates with its remarkable tensile strength of 67.63 MPa.

Keywords: adhesive, steel, surface treatment, cold plasma, wetting, tensile strength

Li-ION battery cell bonding technology

F. Tajti [1*], M. Berczeli [1], Z. Weltsch [2]
[1] Department of Innovative Vehicles and Materials, John von Neumann University, Kecskemét, Hungary
[2] Department of Road and Rail Vehicles, Széchenyi István University, Győr, Hungary

Abstract:

Li-ION battery cells can be used to make battery packs, either for vehicles or to power small electrical household appliances. In battery packs, Li-ION cells are assembled into units and then connected together. The batteries used for interconnection must secure the cells and ensure current conduction. The preparation of the necessary electrical and fastening joints presents a few challenges, including the joining of multiple materials of different thicknesses, possible damage (thermal, mechanical or vibration) during the joining process. Our research will explore the possibilities of different joining technologies that can be used to interconnect Li-ION cells. Resistance welding is the most common joining method, but we are also investigating the feasibility of laser beam welding, soldering and electrically conductive adhesives. In order to achieve adequate mechanical strength, the surface treatment of the workpieces to be joined is investigated, using conventional and high energy density technologies. Furthermore, we investigate the resistance and thermal properties under load of the joints created (Figure 1).

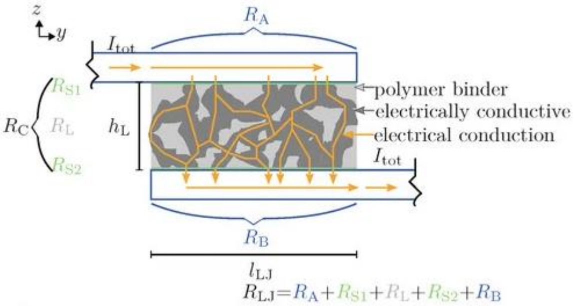


Figure 1: The figure illustrates the local resistances and the determination of the total resistance of the joint during the application of the conductive adhesive.

The use of electrically conductive adhesives, which is easy to automate and does not require expensive machinery, may be suitable for the preparation of battery bonds of smaller size and power. However, surface treatment of the surfaces to be bonded is necessary to increase strength and reduce electrical resistance (Figure 2).

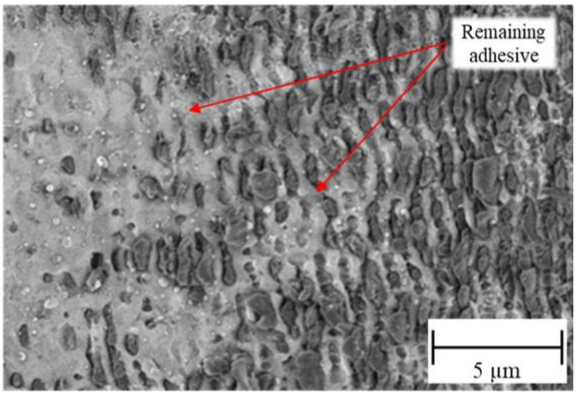


Figure 2: The picture shows the surface structure on a steel plate created by the laser beam surface treatment, and it can be observed that the adhesive is embedded in the surface structure.

Keywords: electrically conductive adhesive, LI-ION, surface treatment, LIPSS, plasma beam surface treatment, laser beam surface treatment, surface structure.

References:

1. Zwicker. M.F.R., Moghadam M, Zhang W, Nielsen C.V, Automotive battery pack manufacturing – a review of battery to tab joining, *Journal of Advanced Joining Processes*, Volume 1, 2020, 100017

Tribology 2024 Session III. A: Lubricants and hydrodynamic lubrication / Biotribology

Recent Advances and Current Challenges in Developing Predictive Models and Design Tools in Tribology

D. Dini [1,*]

[1] Department of Mechanical Engineering, Imperial College London, South Kensington Campus, Exhibition Road, London SW7 2AZ, UK

*Corresponding author: d.dini@imeperial.ac.uk

Abstract:

This talk will cover recent advances in modelling aspects of a variety of problems where the behaviour of tribological interfaces controls the performance of engineering systems. It will start with an overview of the modelling tools developed to study interfacial phenomena across the scales (e.g. [1-5]) and will then move onto demonstrating how in-silico experiments can be used to shed light on various physical, chemical and mechanical phenomena that affect frictional interactions and engineering performance in several applications in the energy sector, including electric vehicles (EVs) and triboelectric nanogenerators (TENGs), biomedical applications, food, consumer goods, as well as functionalized and hierarchical materials and surfaces (e.g. [6-9]).

The main themes discussed are the study of the influence of molecular processes on frictional, rheological and material response, the potential breakdown of continuum theories at the nano- and microscales, as well as important aspects linked to the development of computational models which are twinned with relevant to applications. Many systems involve two or more interlinked phenomena that are governed by mechanisms originating at different scales, for which complex multiscale and multiphysics models are needed. These are still challenging to develop and use as they require multidisciplinary expertise and collaborative effort. A few successful examples portraying how predictive models can be used to better the design of surfaces and materials are provided in this talk, which show how to conduct successful virtual experiments and their necessary links to laboratory tests.

The presentation will conclude with highlighting the current limitations of existing models and techniques and an outlook at future trends and examples of how to change current engineering practice and place tribological interfaces at the centre of design strategy using a bottom-up approach. This will enable to directly use the insight gained from simulations at the smallest scales for the development of new disruptive solutions and to accelerate screening of new material ans lubricant formulations.

Keywords: computational methods, multiscale modelling, multiphysics, friction, lubrication, energy, materials, biomedical applications.

References:

1. Ntioudis, S., JP Ewen, D Dini, CH Turner, A hybrid off-lattice kinetic Monte Carlo/molecular dynamics method for amorphous thin film growth, *Computational Materials Science* 229, 112421, 2023.
2. Ardah, S., FJ Profito, T Reddyhoff, D Dini, Advanced modelling of lubricated interfaces in general curvilinear grids, *Tribology International* 188, 108727, 2023.
3. Ciniero, A., G Fatti, M Marsili, D Dini, MC Righi, Defects drive the tribocharging strength of PTFE: An ab-initio study, *Nano Energy* 112, 108502, 2023.
4. Xu, Y., D Balint, C Greiner, D Dini, On the origin of plasticity-induced microstructure change under sliding contacts, *Friction* 11, 473-488, 2023.
5. Bonari , J., M Paggi, D Dini, A new finite element paradigm to solve contact problems with roughness, *International Journal of Solids and Structures* 253, 111643, 2022.
6. Zhang, X., M Scaraggi, Y Zheng, X Li, Y Wu, D Wang, D Dini, F Zhou, Quantifying Wetting Dynamics with Triboelectrification, *Advanced Science* 9, 2200822, 2022.
7. Eder, S.J., PG Grützmacher, MR Ripoll, C Gachot, D Dini, Does speed kill or make friction better?—Designing materials for high velocity sliding, *Applied Materials Today* 29, 101588, 2022.
8. Weiand, E., F Rodriguez-Ropero, Y Roiter, PH Koenig, S Angioletti-Uberti, D Dini, JP Ewen, Effects of surfactant adsorption on the wettability and friction of biomimetic surfaces, *Physical Chemistry Chemical Physics* 25 (33), 21916-21934, 2023
9. B Kew, M Holmes, E Liamas, R Ettelaie, SD Connell, D Dini, A Sarkar, Transforming sustainable plant proteins into high performance lubricating microgels, *Nature Communications* 14 (1), 4743, 2023.

Tribological behaviours of additively manufactured CoCrMo in bovine calf serum for human joint replacement: understanding the role of process parameters and porosity

Q. Shi [1,*], M.S. Abd Aziz [2,3*], T. Reddyhoff [2], C. Myant [1]
[1] Dyson School of Design Engineering, Imperial College London, London, United Kingdom
[2] Department of Mechanical Engineering, Imperial College London, London, United Kingdom
[3] Fakulti Teknologi Dan Kejuruteraan Mekanikal, Universiti Teknikal Malaysia Melaka, Melaka, Malaysia

Abstract:

CoCrMo alloys are widely utilized in medical implants, such as hip, knee, and shoulder replacements, owing to their superior wear and corrosion resistance. The adoption of additive manufacturing, such as selective laser melting (SLM), has revolutionized the production of joint replacement implants, allowing for the creation of intricate geometries and overcoming the limitations of traditional machining.

While previous studies have identified unique microstructural characteristics and the presence of pores in some SLM alloys, and resultant influences on the lubricating and wear behaviours, SLM CoCrMo is less studied. [1,2] The durability and biocompatibility of implants are paramount, as the degradation of metal materials due to wear in artificial joints can potentially trigger adverse physiological reactions in patients.

Despite the importance of this issue, the understanding of the tribological performance of SLM CoCrMo alloys in biological environments is lacking. This study addresses this gap by examining the influence of porosity generated through varied SLM process parameters on the tribological behaviors of CoCrMo in a biological environment. Measurements of surface roughness, wettability, and hardness aim to unravel the intricate role of pores in tribological behaviors. Subsequent post-tribology analysis provides valuable insights into the underlying tribological mechanisms, shedding light on factors contributing to the degradation and performance of SLM CoCrMo under lubricated conditions.

This research contributes to advancing our understanding of the tribological behaviors of SLM CoCrMo alloys, offering crucial insights for the enhancement of joint replacement applications.

Keywords: Selective laser melting, CoCrMo, joint replacement, biotribology, tribological behaviours wettability, bovine calf serum, friction, lubrication

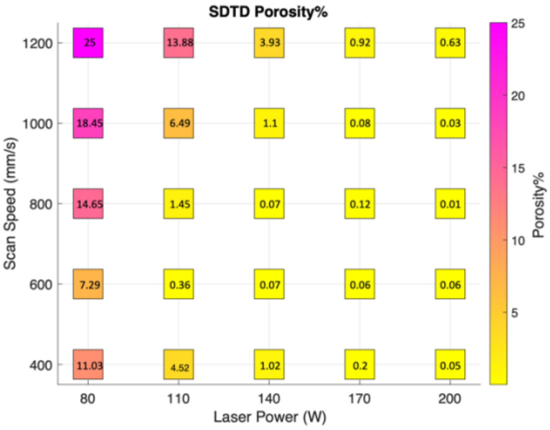


Figure 1: Figure illustrating a matrix of the porosity percentages of SLM CoCrMo addtively manufactured with varied scan speeds and laser power.

References:

1. Zhu, Yi, et al. "Material characterization and lubricating behaviors of porous stainless steel fabricated by selective laser melting." Journal of Materials Processing Technology 262 (2018): 41-52.
2. Li, Hua, et al. "Effect of process parameters on tribological performance of 316L stainless steel parts fabricated by selective laser melting." Manufacturing letters 16 (2018): 36-39.

PVD nitrides to be used for increased life time of tools and components

P.H. Mayrhofer*
Institute of Materials Science and Technology, TU Wien, A-1060 Wien, Austria

Abstract:
For mechanically dominated load profiles, nitrides are preferred, while oxides offer better protection against high-temperature corrosion. Combined mechanical and thermal loads therefore call for nitrides with excellent temperature and oxidation resistance. How to develop such nitrides with excellent strength and toughness combined with exceptional thermal stability is the focus of this talk.

Using transition metal nitride coatings, we will discuss important guidelines for material development to improve strength, fracture toughness, and stability. In particular, the stability (emphasis on phase stability to composition and temperature, but also to oxidation) of nitrides is a highly interesting task. We will discuss various model systems to explore the possibilities of alloy and structural developments.

The concept of high entropy is also very beneficial for hard ceramic thin films. We will see that, for example, (Hf,Ta,Ti,V,Zr)N and (Al,Cr,Nb,Ta,Ti)N easily outperform their commonly used binary or ternary constituents in terms of thermal stability and thermomechanical properties. In addition, all of the highly entropic ceramic sublattice thin films studied were relatively insensitive to variations in deposition parameters – which is good because their properties are at a high level.

The individual concepts allow the materials to be designed to meet the ever-growing demand for further coatings tailored to specific applications.

Keywords: PVD, protective coatings, thermal stability, oxidation resistance, hardness, fracture toughness, protective applications.

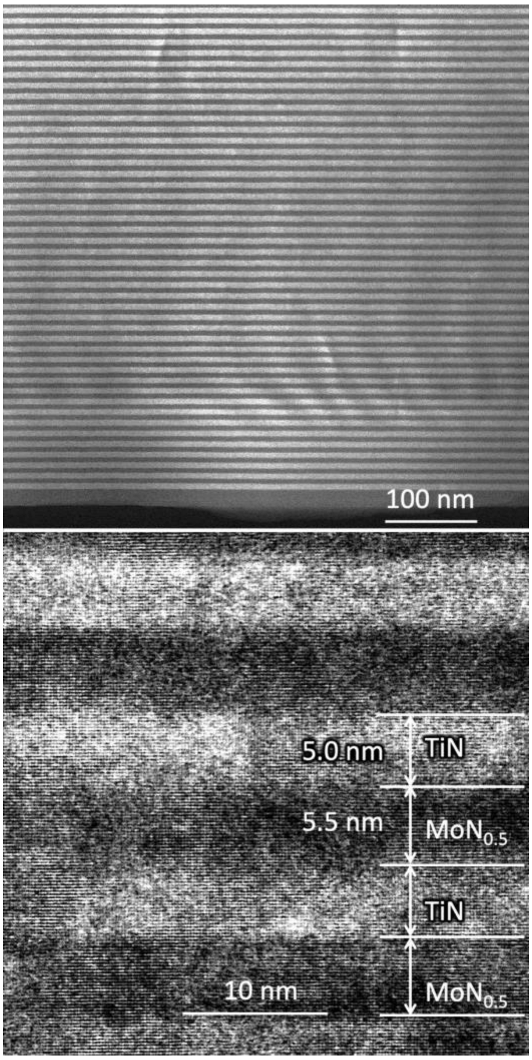


Figure 1: Cross-sectional TEM investigations of a 001-oriented single-crystalline TiN/$MoN_{0.5}$ superlattice with Λ = 10.5 nm, grown on MgO(001). Adapted from [1].

References:

1. Z. Gao, J. Buchinger, N. Koutná, T. Wojcik, R. Hahn, P.H. Mayrhofer, , Acta Mat. 231 (2022) 117871 1–12

Insight into the fundamental behaviour of Organic Friction Modifiers doped with small polar molecules

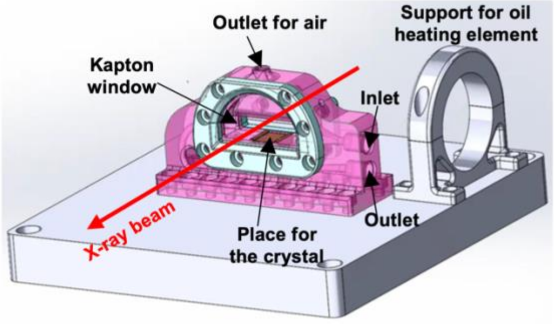
I. Kicior[1,2,*], T. Poręba[1], E. A. Willneff[3], J. Frey[1], V. Honkimäki[1], A. Morina[4], P. Dowding[5],
S. L. M. Schroeder[2]
[1] Experiments Division, European Synchrotron Radiation Facility, Grenoble, France
[2] School of Chemical and Process Engineering, University of Leeds, Leeds, UK
[3] School of Design, University of Leeds, Leeds, UK
[4] School of Mechanical Engineering, University of Leeds, Leeds, UK
[5] Infineum UK Ltd., Milton Hill, UK

Abstract:

Organic Friction Modifiers (OFMs) are a class of lubricant additives with surfactant-like structure, with usually at least 12 carbon atoms in the hydrophobic chain and a polar head group that change their adsorption behaviour[1]. These additives have been known for a century but became less popular after the discovery of zinc dialkyl dithiophosphates (ZDDPs). ZDDPs are now a subject of sustainability concerns due to the emission of sulphur and phosphorus compounds during their degradation. OFMs do not contain harmful elements, are biodegradable and are therefore studied as a more sustainable alternative. The common theory behind the operation of OFMs is that they adsorb to the surface as a monolayer and physically separate the contact in the boundary lubrication regime. However, recent studies, both simulation and experimental, suggest that OFMs form more complex structures, e.g. clusters of reversed micelles, and their action depends on the friction conditions and presence of othr molecules[2,3], especially small polar dopants in the lubricant by design or under vehicle operation conditions. To understand the behaviour of OFMs, structural studies *in situ*/*operando* are required. Important work has been done with neutron techniques[4], but these are relatively slow and may require deuteration to enhance contrast. Therefore, we apply synchrotron X-ray scattering techniques that offer high photon flux, enabling fast measurements and good spatial resolution even for weakly scattering samples. 3 chosen OFMs basen on oleyl chain (18 carbon atoms, monounstaturated) have been studied in the model base oil *n*-dodecane, with and without water/acetic acid doping. Mini Traction Machine tests and laboratory XPS (X-ray photoelectron spectroscopy) are compared with *ex situ* SAXS and XRR (small angle X-ray scattering, X-ray reflectivity) and *in situ* XPDF (X-ray pair distribution function) and SAXS in conditions of shear, temperature and pressure.

Keywords: organic friction modifiers, glycerol monooleate, adsorption, X-ray scattering, lubricant additive, mini-traction machine, X-ray photoelectron spectroscopy, synchrotron, *n*-alkane, micelles, polar dopant.

Figure 1: Schematic of custom-built XRR flow cell that allows for the in situ studies of OFM adsorption to the single crystal under flow conditions. Temperature and flow speed can be controlled.



References:

1. Spikes, H. (2015) Friction Modifier Additives, *Tribol. Lett.*, 60, 5.
2. Bradley-Shaw, J., Camp, P., Dowding, P., Lewtas, K. (2016) Molecular Dynamics Simulations of Glycerol Monooleate Confined between Mica Surfaces, *Langmuir*, 32, 31, 7707-7718.
3. Armstrong, A., Apóstolo, R., McCoy, T., Allen, F., Doutch, J., Cattoz, B., Dowding, P., Welbourn, R., Routh, A., Camp, P. (2024) Experimental and simulation study of self-assembly and adsorption of glycerol monooleate in *n*-dodecane with varying water content onto iron oxide, *Nanoscale*, 16, 1952-1970.
4. Armstrong, A., McCoy, T., Welbourn, R., Barker, R., Rawle, J., Cattoz, B., Dowding, P., Routh, A. (2021) Towards a neutron and X-ray reflectometry environment for the study of solid-liquid interfaces under shear, *Sci. Rep.*, 11, 9713.

Ecolubricants based on vegetable oils with ionic liquid

M.D. Avilés[1*], P. Mostaza-Ucedo[1], T. Caparrós [1] and F.J. Carrion-Vilches[1]

[1]Grupo de Ciencia de Materiales e Ingeniería Metalúrgica. Universidad Politécnica de Cartagena
C/ Doctor Fleming sn, 30201 Cartagena (SPAIN)

Abstract:

There is increasing interest in vegetable oils for applications in industrial lubrication rather than mineral oils. Vegetable oils are biodegradable and come from natural resources; with null toxicity in comparison with mineral oils derived mainly from petroleum. Nevertheless, the properties of these natural oils must be modified to improve their thermal stability and resistance to oxidation, in order to extend the service life.

The tribological properties of vegetable oils, such as the friction coefficient, wear resistance, or load-carrying ability, need to be improved to make them suitable for high-performance applications. To achieve this requires the development of new lubricant formulations that include chemical modification as well as the use of additives. The ecolubricants obtained will have a minimal environmental impact and better tribological performance, thereby making them a more attractive alternative to mineral oils.

Ionic liquids (IL), a new class of compounds, have demonstrated their potential as neat lubricants or as additives for lubricants in a variety of types of contacts and configurations, due mainly to the polar nature of IL molecules. This ability makes it possible to develop tribo-layers on metal surfaces and diminish the friction and surface damage [1-2]. Furthermore, other properties such as non-flammability, non-volatility, high thermal-oxidative stability, and high thermal conductivity support IL use in tribology as eco-friendly lubricants.

Although the addition of IL to vegetable oils has scarcely been studied, the conjunction of the triglycerides and fatty acids contained in vegetable oils and the IL, enables the formation of tribofilms on metallic surfaces. This protects the surface from damage and reduces friction, and it is the key for the formulation of potential new biolubricants [3-4].

The present communication shows the tribological properties of neat and epoxidized avocado oils in comparison to those with the addition of IL diethylmethylammonium methanesulfonate. Friction coefficients and wear rates were studied with a pin-on-disc tribometer using a tribopair of sapphire/stainless steel at 1.4 GPa of Hertz contact pressure. The friction coefficient and wear rate in the case of epoxidized avocado oil (EAv) with 1 wt.% of IL showed an important reduction (Figure 1). Optical and Electronic microscopy, and different spectrocospic techniques were used to establish wear mechanisms and the stability of the oils in tribological conditions.

Keywords: ecolubricant, vegetable oil, epoxidized oil, avocado oil, ionic liquids, tribology

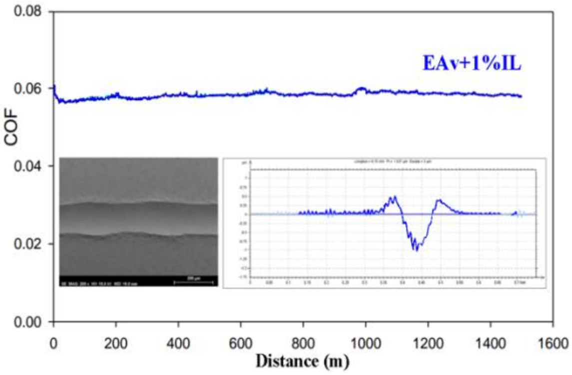


Figure 1: Friction coefficient and wear track for the tribopair sapphire/stainless steel using a vegetable oil base with the addition of ionic liquid.

References:

1. M.D. Bermúdez, A.E. Jiménez AE, J. Sanes, FJ Carrion. (2009), Ionic liquids as advanced lubricant fluids. *Molecules* 14, 2888-2908.
2. I. Minami. (2009), Ionic liquids in tribology.*Molecules* 14, 2286–2305.
3. R. Gusain, O.P Khatri. (2016),. Fatty acid ionic liquids as environmentally friendly lubricants for low friction and wear. *RSC Advances* 5, 3462-3469.
4. K.C. Wickramasinghe, H. Sasahara, E.A. Rahim, G.I.P. Perera. (2021), Recent advances on high performance machining of aerospace materials and composites using vegetable oil-based metal working fluids. *Journal of Cleaner Production*, 310, 127459.

Optimizing Tribological Performance: Exploring the Role of Surface Texture and Lubricants Composition

M.S. Abd Aziz [1,2,*], T. Reddyhoff [1], Chiharu Tadokoro [3]
[1] Department of Mechanical Engineering, Imperial College London, London, United Kingdom
[2] Fakulti Teknologi Dan Kejuruteraan Mekanikal, Universiti Teknikal Malaysia Melaka, Melaka, Malaysia
[3] Department of Mechanical Engineering, Saitama University, 255 Shimo-Okubo, Sakura, Saitama 338-8570, Japan

Abstract:

Introducing surface texture to a sliding component has been shown to effectively improve tribological performance of a machine element, specifically in terms of reducing friction and wear. Thus, there has been a surge of need on the study of textured surfaces, often focusing on the influence of geometrical texture characteristics towards coefficient of friction (Zhang et al., 2017). However, lubricant composition has been a relatively understudied area and should be of an importance in view of its influence on the performance of surface texture (Wang et al., 2020). To address this research gap, our current study delves into the comparison of the friction-reducing effectiveness of surface-textured components versus non-textured references across a range of commercial and model lubricant compositions. The findings demonstrated that not only did potential optimization of lubricant for textured surface affects the efficiency of a machine element, but also provide insights into how specific additives interact with textured features, thereby illuminating its underlying mechanisms. As a result, the components influencing the observed tribological behaviors are enlightened, providing valuable insights for the advancement of effective friction-reducing strategies in engineering applications.

Keywords: Surface Texturing, Lubricants, Additive

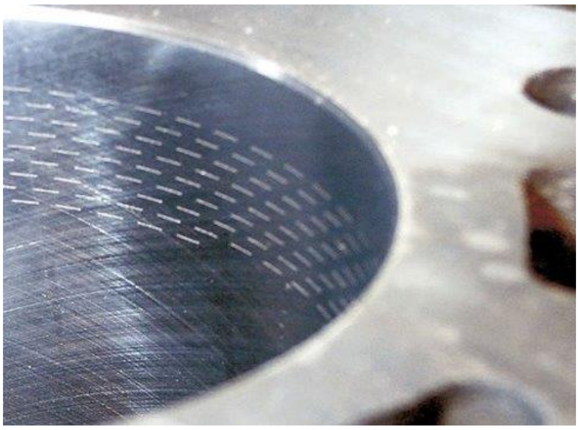


Figure 1: Figure illustrating the surface texturing application towards mechanical component.

References:

1. Wang, J., Yan, Z., Fang, X., Shen, Z., & Pan, X. (2020). Observation and experimental investigation on cavitation effect of friction pair surface texture. *Lubrication Science*, *32*(8), 404-414.
2. Zhang, S., Zeng, X., Igartua, A., Rodriguez-Vidal, E., & Van der Heide, E. (2017). Texture design for reducing tactile friction independent of sliding orientation on stainless steel sheet. *Tribology letters*, *65*, 1-11.

A bearing test stand to represent E-VTOL air-screw loads in vertical and forwards flight.

R. T. Wragge-Morley [1*], G. C. Barnaby[1], J. M. Yon[1] and P. H. Mellor
[1] School of Electrical, Electronic and Mechanical Engineering, University of Bristol UK

Abstract:
The aerospace sector is currently seeing a rapid growth of interest in electrically propelled light aircraft for use as air-taxis, public service vehicles or private craft [1]. Many of these vehicles fall into either the Electric Vertical Take-Off and Landing (E-VTOL) or tilt-rotor categories. These types of multi-rotor craft can impose very severe loading cases on their propulsion machinery. Many present designs mitigate the direct mechanical loading of electrical machine components by inclusion of a gearbox, but this adds both mass and complexity. The difficulty arises from the use of fixed-pitch fans for small distributed propulsion craft. These induce a cyclical combined axial, radial and bending moment about the shaft end at the blade pass frequency when vertically oriented in forwards flight. This phenomenon is known as blade 'flapping' and the advancing blade can generate up to 80% of the lift [2]. When compared to a conventional rotorcraft, the fan speeds for distributed propulsion craft are significantly higher, and typically no feathering mechanism is fitted to control blade 'flapping'. Most bearing designs are arranged to support high loading in either axial or radial directions and solutions for combined loading tend to add either mass or friction. As a result, the majority of bearing test machines focus on loading in a single direction [3] although solutions for combined axial and radial loads [4] and moment loading exist [5].

To fully reproduce the loading experienced by the drive end bearings of an E-VTOL propulsion machine with a directly coupled fixed-pitch propellor would require a loading system with five degrees of freedom, the sixth being shaft rotation. Additionally the bandwidth of actuation would need to be able to accommodate the blade pass frequency in order to replicate cyclical loading. In this study, we present a viable simplification that allows a combined radial and thrust bearing set to be loaded cyclically with combined forcing. The mechanical layout and actuation strategies of such a test-stand, illustrated in Figures 1 and 2, will be discussed, including the possibility of using novel actuation techniques to increase bandwidth [6]. An overview will also be given of the development of an instrumentation system capable of generating training data for prognostics.

Keywords: Wear and friction, Contact mechanics, Test methodologies and measurement technologies, Rotorcraft, Rolling Element Bearings.

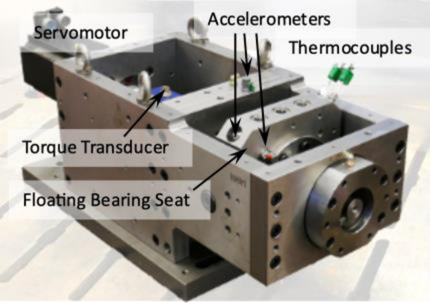


Figure 1: Main chassis of bearing test stand.

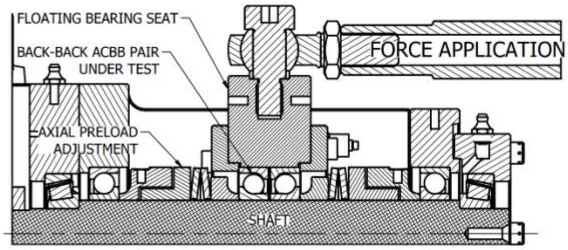


Figure 2: Sectional schematic of bearing loading system.

References:

1. Osita Ugwueze, Thomas Statheros, Mike A. Bromfield and Nadjim Horri, "Trends in eVTOL Aircraft Development: The Concepts, Enablers and Challenges," in AIAA SCITECH 2023 Forum, National Harbor MD, USA, 2023.
2. John Seddon and Simon Newman, Basic Helicopter Aerodynamics Third Edition, Chichester UK: John Wiley and Sons Ltd., 2011.
3. T. A. Harris, Rolling Bearing Analysis, Fourth Edition, Wiley-Interscience, 2001.
4. J J Blake and C E Truman, "Measurement of running torque of tapered roller bearings," Proceedings of the Institute of Mechanical Engineers Part J, Journal of Engineering Tribology, vol. 218, pp. 239-249, 2004.
5. Sanket Yadav, Hagen Elgeti, "Elgeti Engineering Articles 2019-10 Bearing Methods and Strategies," October 2019.

[Online]. Available: https://www.elgeti-engineering.de/wp-content/uploads/2020/10/Publication-2019-10-Bearing-testing-methods-and-strategies_V8.pdf. [Accessed 22 04 2022].

6. Wragge-Morley, R. T., Yon, J. M. & Mellor, P. H.,, "Design of a Piezo-Electric Actuator for Dynamic Testing of Rolling Element Bearings," in Proceedings ICEM20: Experimental Mechanics in Engineering and Biomechanics, Porto, 2023.

SICT 2024 / Plasma Tech 2024 Session III. B: Coatings for Energy and Environmental Applications Plasma application in Energy and environment

Non-thermal plasma: an efficient technology for regeneration of coked zeolite

Ludovic PINARD and Catherine BATIOT DUPEYRAT
Institut de Chime des Milieux et Matériaux de Poitiers,IC2MP,UMR-7285,France.

Abstract:

The main cause of acidic zeolite deactivation is carbon deposition (coke), which poisons or blocks the accessibility of reactants to active sites. Such deactivation and the associated regeneration of catalysts represent a major financial burden and important environmental costs for industrial processes. Coked catalysts can be totally regenerated by burning-off and the coke molecules trapped in micropores (10-5 Å) begins to be oxidized from temperatures (T_{ox}) higher than 600 K, while complete regeneration requires T_{ox} above 800 K. The regeneration at high temperature can lead to irreversible damage of the zeolite structure. The off-site regeneration (OSR) of spent catalysts is often economically more interesting than using fresh catalysts. OSR is further supported by industry due to safety regulations and time issues, and furthermore, allows the development of eco-friendly and energetic efficient processes.

We have recently shown that coke combustion assisted by non-thermal plasma (NTP) is a valuable alternative to classical thermal treatments regardless of the reactor geometry (pin to plate [1-2], fixed bed Barrier discharge reactor [3-4] allowing for total regeneration of zeolite catalysts at room temperature.

The regeneration of a series of coked zeolites, including FAU, MOR and MFI framework, was investigated using a DBD plasma reactor. It has been observed that the efficiency of coke oxidation by non-thermal plasma is very sensitive to both the coke nature and its toxicity. Coke precursors constituted of one or two aromatic rings are totally oxidized into CO_2, while polyaromatic molecules yield organic volatile compounds. For MFI and FAU zeolites with three-dimensional 10 or 12 membered ring channel systems, coke toxicity is moderate. As a consequence, the short-lived oxygenated species generated by NTP can access and oxidize coke molecules. The kinetic oxidation rate is independent of the size of the pore aperture.

As the partial kinetic order for coke is zero the time required to regenerate the zeolite (recovery of the initial micropore volume and Brønsted acidity) is dependent on the initial coke content.

For MOR, featuring a one-dimensional 12 membered ring channel system, coke toxicity is very high, as one coke molecule located on the pore mouth blocks the access to the entire pore channel. As a result coke removal rate is slower and even prolonged regeneration times do not permit for full recovery of microporosity and acidity.

Compared to classical calcination for the regeneration of spent catalysts, non -thermal plasma reveals as cleaner technology

Keywords: zeolite, DBD, regeneration

References:

1. L. Jia, L. Al Farouha, L. Pinard, S. Heden, J. –D Comparot, K. Ben Tayeb, A. Dufour, , H. Vezin, C. Batiot-Dupeyrat "New routes for complete regeneration of coked zeolite" *Appl. Catal. B: Environ*. 291 (2017) 82-91.
2. L. Pinard, N. Ayoub, C. Batiot-Dupeyrat, "Regeneration of a coked zeolite via nonthermal" plasma process: a parametric study" *Plasma Chemistry and Plasma Processing*, 39 (2019) 929-936
3. A.Astafan, C. Batiot Dupeyrat, L. Pinard, "Mechanism and Kinetic of Coke Oxidation by Nonthermal Plasma in Fixed Bed Dielectric Barrier Reactor" *J. Phys. Chem. C 123 (2019) 9168-9175*
4. A. Astafan, A.Sachse, C. Batiot Dupeyrat, L. Pinard, "Impact of the framework type on the regeneration of coked zeolites by non-thermal plasma in a fixed bed dielectric barrier reactor" *Catalysts* 9 (2019) 985-994

Preparation of Nanoparticulate WO_3/MoO_3 Films for Making Electrochromic and Energy-Storage Devices

Wen-Bin Jian*, Chia-Chun Wei, Po-Hung Lin, Tung-Han Wu, Jun-Wei Huang
Department of Electrophysics, National Yang Ming Chiao Tung University, 1001 University Road,
Hsinchu 30010, Taiwan

Abstract:

Sustainable developments to improve energy consumption in buildings and energy storages are essential issues that can be solved by using green technologies. In this talk, we present electro-exploding wire and spray coating techniques for the synthesis of nanoparticulate films of WO_3-MoO_3 composite films. The nanoparticulate films are used to assemble electrochromic devices for the transmittance and energy storage measurements. The nanoparticulate WO_3-MoO_3 films exhibit high electrochromic performances with a diffusion coefficient of deintercalation of $3.39X10^{-10}$ cm^2/s, a charge density of 28.5 mC/cm^2, the coloration efficiency of 40.9 cm^2/C at the light wavelength of 430 nm, and the transmittance variations of about 17% and 44% at wavelengths of 430 and 635 nm [1,2].

The nanoparticulate WO_3-MoO_3 films not only display good electrochromic performances but also energy storage capabilities. These films exhibit a high porosity that is beneficial for ion diffusions in electrolyte and for increasing the efficiency of oxidations and reductions. In particular, the porous nanoparticulate WO_3-MoO_3 films can be stacked to increase the thickness and to enhance the energy storage capability.

Keywords: protein folding, nanoporous sol-gel glasses, silica-based biomaterials, circular dichroism spectroscopy, surface hydration, crowding effects, micropatterning, biomedical applications.

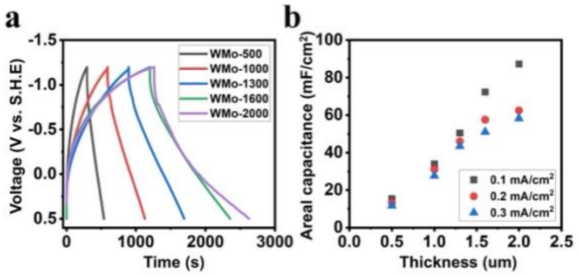


Figure 1: (a) Galvanostatic charge-discharge (GCD) curves at a current density of 0.1 mA/cm2 for Samples WMo-500, WMo-1000, WMo-1300, WMo-1600, and WMo-2000. The charge-discharge operation voltage is set in the range between -1.2 and +0.5 V. (b) Calculated areal capacitance from GCD curves as a function of film thickness. The black squares, red dots, and blue triangles represent areal capacitance measured at current densities of 0.1, 0.2, and 0.3 mA/cm2, respectively.

References:

1. Wei, C. C., Wu, T. H., Huang, J. W., Young, B. L., Jian, W. B., Lin, Y. L., Chen, J. T., Hsu, C. S., Ma, Y. R., Tsukagoshi, K. (2023), Nanoparticulate films of WO_3 and MoO_3 composites for enhancing UV light electrochromic transmittance variation and energy storage applications, *Electrochimica Acta* 442, 141897 (2023).
2. Chang, C. M., Chiang, Y. C., Cheng, M. H., Lin, S. H., Jian, W. B., Chen, J. T., Cheng, Y. J., Ma, Y. R., and Tsukagoshi, K. (2021), Fabrication of WO_3 electrochromic devices using electro-exploding wire techniques and spray coating, *Sol. Energy Mater. Sol. Cells* 223, 110960.

Room Temperature Synthesis of Hydrogen Permeation Barrier for Storage and Transportation Application

S. R. Brankovic[1], N. Amiri[1], I. Rabbi[1].
[1] Cullen College of Engineering, University of Houston, Houston TX 77204, USA

Abstract:

Distribution of hydrogen in future hydrogen-based economy requires a dense pipeline network for its safe delivery. Natural take is to consider the existing networks of natural gas (NG) infrastructure. However, the main issue is that hydrogen-embrittlement on NG pipes' steels significantly reduces their fracture toughness and threatens their structural integrity[1]. Besides developing a new grade of steels less susceptible to the embrittlement, effective hydrogen permeation barriers (HPBs) remain important solution for re-purposing of the existing NG infrastructure for hydrogen transport. Besides a few specific metals, candidates are some oxides, carbides, and nitrides. These coatings require extreme conditions for their synthesis, which renders their application impractical.

We employed strategy for HPB synthesis based on electroless Cu-deposition process[2]. We have employ solution chemistries exploring a wide range of reducing agents. These include formaldehyde, glyoxylic acid, Na_2HPO_2 … and others. Variety of complexing agents were considered as well; EDTA, sodium citrate, sodium potassium tartarate and others. Our optimum solution design favored a large driving force for Cu reduction providing high nucleation rates and films with fewer defects and dense grain boundaries. The comparative analysis between hydrogen permeation rates through bare steel samples and steel samples coated with Cu HPB films is performed using *Devanathan - Stachurski* permeation technique[3]. The Cu-HPB thickness varied from experiment to experiment. More than 30 samples have been evaluated. An integral example of these measurements is shown in Figure 1. Permeation reduction factor (PRF) for Cu-HPB as a function of the ratio between steel and Cu-HPB thickness is shown against theoretical calculations. Our results exceed theoretical predictions by factor of 2-5.

Keywords: Hydrogen, hydrogen permeation barrier, hydrogen embrittlement, permeation reduction factor, Cu electroless deposition, hydrogen economy.

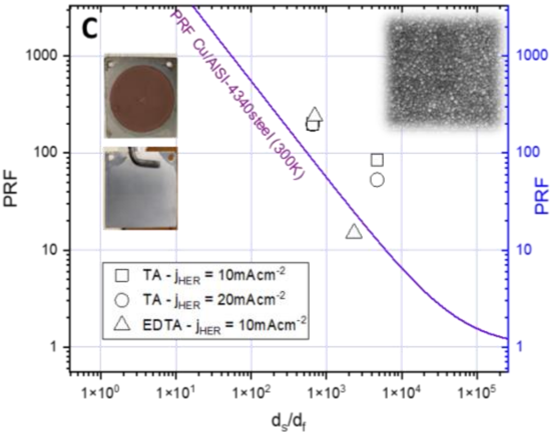


Figure 1: Ccalculations for PRF of Cu/(AISI 4340 steel) as a function of d_s/d_f ratio. Square, circular, and triangular dots represent PRF measurements for Cu-HPB/(ASTM A36 steel) samples produced from tartaric acid (TA) and EDTA solutions. Insets show optical images of steel and Cu/steel samples (left) and SEM of Cu surface morphology (right).

References:

1. V. Nemanic, Hydrogen permeation barriers: Basic requirements, materials selection, deposition methods, and quality evaluation, Nuclear Materials and Energy, 19 (2019) 451-457.
2. M. Paunovic, *Chapter 17: Electroless Deposition of Copper, in Modern Electroplating, fifth Edition*, Eds, M. Schelsinger and M. Paunovic, Willey, NY (2005), p. 433.
3. Iyer, R.N., Pickering, H.V., 1990. Mechanism and kinetics of electrochemical hydrogen entry and degradation of metallic systems. Annu. *Rev. Mater. Sci.* **20**, 299-338.

Film-Based Repair System for Offshore Wind Tower Coatings

T. Marquardt[1*], A.W. Momber[1], S. Buchbach[2], M. Irmer[3], M. Beitmann[4], B. Koeller[5]
[1] Muehlhan Holding GmbH, Hamburg, Germany
[2] Fraunhofer IFAM, Bremen, Germany
[3] Fraunhofer IGP, Rostock, Germany
[4] Coroplast Fritz Müller GmbH & Co. KG, Wuppertal, Germany
[5] RENOLIT SE, Frankenthal, Germany
*Corresponding author; marquardt@muehlhan.com

Abstract:

A new method for protecting the surfaces of offshore wind tower structures is proposed as an alternative to traditional multi-layer spray-applied or brush-applied liquid coatings. A film repair system, consisting of a PMMA-PVC hybrid film and a pressure sensitive adhesive, has been developed for the use on offshore wind towers. This system has several advantages compared to traditional multi-layer organic paint systems. One major advantage is the significantly shorter repair time. The film system can be quickly and easily applied, thereby significantly reducing downtime, and increasing the efficiency of repairs. Additionally, the film is highly durable and resistant to mechanical damages and UV exposure. The repair system is cost-effective, as it requires less resources and labor. Films provide a smoother and more consistent finish compared to paint systems, resulting in a more attractive appearance and a uniform film thickness. The film system is a more sustainable option, as it neither releases volatile organic compounds (VOCs) into the air nor liquid paint material into the water during application, unlike organic paint systems. Overall, the use of a hybrid film and pressure sensitive adhesive for repair of offshore wind towers offers a reliable, environmentally friendly, and cost-effective alternative to traditional paint systems. Accelerated cyclic aging tests according to ISO 12944-9 were conducted to simulate the effects of offshore exposure, including chlorides, condensation, and temperature-(changes), on the performance of the film repair system. UV-weathering over 10,000 hours (ISO 4892-2) was performed, and the mechanical resistance was tested in regards of abrasion (ISO 7784-2), pressure loads, and impact loads (ISO 6603-2). Data acquired from the tests was analyzed using statistical methods, such as regression analysis and ANOVA. The results of the statistical analysis were used to optimize composition and formulation of the system and to improve its performance. The optimized repair system is validated through a mockup during field testing in an offshore environment on Helgoland, North Sea (Fig. 1). Based on the results of the field testing, the system will be refined as needed.

Keywords: OWEA, Coating repair, Film system, PSA, Statistical analysis, FoKO-Wind.

Figure 1: Offshore wind tower mockup structure with predefined repair spots for film application. (Copyright: Muehlhan Holding GmbH)

References:

1. Ren, Z. et al. (2021). Offshore wind turbine operations and maintenance: A state-of-the-art review. DOI: 10.1016/j.rser.2021.110743.
2. Momber, A.W. (2024). Corrosion and Corrosion Protection of Wind Power Structures in Marine Environments. Vols. 1&2, Elsevier, London.

Development of a 100 kW Steam Plasma Torch (SPT) for Waste Remediation and Clean Hydrogen Production

Hamid Reza Yousefi, Bernd Glocker, Steffen Pauly, Helmut Koch
PlasmaAir AG, Weil der Stadt, Germany

Abstract:

We present the development of a robust 100 kW DC arc discharge steam plasma torch designed for extended operational lifetime, reaching up to 200 hours. Operating efficiently with a steam input of 10 kg/h, this innovative system not only emphasizes durability and performance but also highlights its potential for scalability. The integration of steam as a working fluid not only enhances environmental sustainability but also opens avenues for increasing both steam input and power in future iterations.

Furthermore, we delve into the crucial industrial applications, with a particular focus on the versatility of steam plasma. Among its myriad potential uses, we highlight several key applications, including waste treatment and methane reforming. The torch demonstrates remarkable efficiency in treating industrial waste, offering a sustainable solution for hazardous waste treatment. Additionally, one interesting application is the pyrolysis of plastic waste, where the torch can be employed as a heat source in the reactor or for conditioning the pyrolysis gas.

Moreover, the system showcases its versatility in methane reforming processes, where steam plasma, enriched with oxygen radicals and hydrogen, facilitates the efficient conversion of methane into hydrogen and carbon monoxide. This not only positions the torch as a promising solution for clean hydrogen production but also underscores its potential in advancing sustainable energy practices.

In addition to these applications, the torch proves to be a valuable tool for the treatment of typical gas streams in semiconductor industries. The precision and controllability of the plasma make it suitable for addressing the specific needs of semiconductor processing, showcasing its adaptability across diverse industrial sectors.

Additionally, our plasma torch is designed with versatility in mind, allowing seamless integration with large-scale reactors for further testing and optimization. Furthermore, the electrode design in our system features a rod cathode, contributing to the torch's stability and reliability. We also outline our future roadmap, with plans to develop a 250 kW steam plasma torch, promising even greater capabilities. Our presentation will delve into the design, performance characteristics, and nuanced applications of this cutting-edge plasma torch, laying the foundation for further research to explore enhanced steam and power capabilities. Further details will be provided in the full paper.

Keywords: arc discharge plasma, plasma torch, steam plasma torch, waste treatment, methene pyrolysis

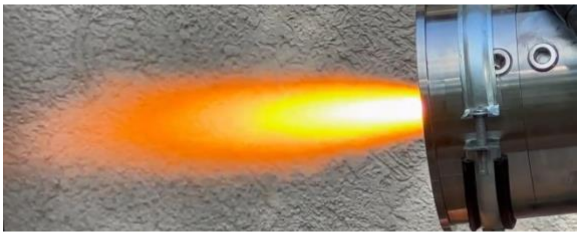


Figure 1: A schematic representation of a 100 kW steam plasma torch, showcasing the system's operation with a steam input of 10 kg/h

References:

1. Smith, J., et al. (2022). "Innovations in Steam Plasma Technology: A Comprehensive Review." Journal of Advanced Plasma Science, 15(3), 120-135.
2. Greenfield, A., & Williams, R. (2021). "Advancements in Waste Treatment Technologies: A Case Study on Plasma Torch Applications." Environmental Engineering Journal, 28(2), 75-89.
3. Patel, M., et al. (2020). "Steam Plasma Reforming of Methane for Sustainable Hydrogen Production: Experimental Insights." International Conference on Plasma Science and Applications, Proceedings, 45-52.

Atomic Scale Loading of Palladium on Oxygen-Deficient Cerium Oxide for Electrocatalytic Applications

A.B. Yousaf[1*], P. Kasak[1*]
[1]Center for Advanced Materials, Qatar University, Doha 2713, Qatar

Abstract:

The design and development of portable power source devices at a larger scale to mitigate the energy crises for next generation modern communities have drawn huge attention over decades. Among those, fuel cells have long been considered as producers of green and sustainable energy [1-3]. Therefore, the application and broadening of fuel cell research and portable device fabrication for the sake of green energy are highly demanded. As the catalyst materials play a role of pillar in these renewable energy devices, herein, we have designed to develop facile and effective strategies for the fabrication of effective catalyst materials comprising metal oxide loaded with ultra-low atomically dispersed metals active sites (noble metals species) for the construction of scalable FCs electrode. The cerium oxide support has been extensively used to fabricate the Pd on its surface. The atomically dispersed Pd species were characterized by means of physical characterizations such as X-ray diffraction, X-ray photo-electron microscopy, and high-resolution transmission electron microscope. The as-measured structural characterizations successfully proved the synthesis of the desired catalyst material. Later on, the catalyst was tested for electrocatalytic methanol oxidation reactions (MOR), and the performance exhibited expected trends of MOR from support to the atomically dispersed Pd on its surface.

Keywords: atomic scale loading, palladium, cerium oxide, oxygen-deficient support, electrocatalysis, methanol oxidation reactions, fuel cells

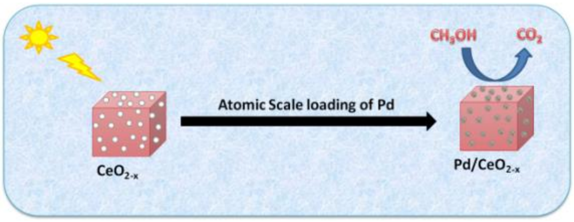


Figure 1: Figure illustrating the development of the electrocatalyst and its application purpose.

Acknowledgements

Research reported in this publication was supported by the Qatar Research Development and Innovation Council ARG01-0524-230315. The content is solely the responsibility of the authors and does not necessarily represent the official vies of Qatar Research Development and Innovation Council.

References:

1. Zhang, J., Yang, H., Liu, B. (2020) *Adv. Energy Mater.*, 11, 2002473
2. Zhao, D., Zhuang, Z. W., Cao, X., Zhang, C., Peng, Q., Chenand, Li, Y. D., (2020) *Chem. Soc. Rev.*, 49, 2215–2264.
3. Kodama, K., Nagai, T., Kuwaki, A., Jinnouchi, R., Morimoto, Y., (2021) *Nat. Nanotechnol.*, 16, 140–147.

Novel deposition technique of α-Al2O3-based coating as a hydrogen permeation barrier

N. Laadel [1,2,*], M. El Mansori [1,3], N. Frederich[2], S. Marlin[2]
[1] MSMP, Arts et Métier Institute of Technology, Aix-en-Provence, France
[2] Saint-Gobain Research Provence, Cavaillon, France
[3]Texas A&M Engineering Experiment Station, Institute for Manufacturing Systems, Texas, USA

Abstract:
Hydrogen-induced cracking of metals or hydrogen embrittlement (HE) is a well-known surface-related phenomenon. Coating deposition using ceramic materials with specific properties to resist hydrogen permeation is among the promising solutions[1]. This work addresses a novel wet deposition technique, namely the electrophoretic deposition (EPD), to fabricate aluminum oxide coatings onto metallic substrates. This process is based on the movement and deposition of charged particles under an electric field onto a conductive electrode to deposit thin ceramic layers. Compared to other advanced techniques, the EPD offers several advantages such as formation time, simple setup, and apparatus, as well as fewer restrictions in terms of substrate shape and geometry[2]. The EPD process is optimized towards its potential use for processing of hydrogen barrier coatings. Thus, the main objective of this work is to achieve compact alumina layers with as little pores and cracks as possible, showing good adhesion. The deposition of α-Al2O3 coating has been chosen due to the intrinsic low permeability of this material, which is considered as a good hydrogen permeation barrier (HPB) candidate[3]. The coating properties (e.g., thickness, uniformity, and porosity) are adjusted by controlling the current intensity during the treatment, the suspension stability, and the deposition time. The samples were analyzed comprehensively using scanning electron microscopy, x-ray diffraction and image analysis to assess the quality of the coatings and to develop a detailed understanding of the processes involved during layer formation. The results show that the coating quality can be improved significantly by using proper solvent with high suspension stability. The quality of the coating depends as well to the use of a proper sintering temperature. The resulting optimized coating delivers uniform and homogeneous α-Al2O3 layers showing low porosity with good adhesion to the substrate.

Keywords: hydrogen embrittlement, hydrogen permeation barrier, alumina coating, electrophoretic deposition, hydrogen storage and transportation applications.

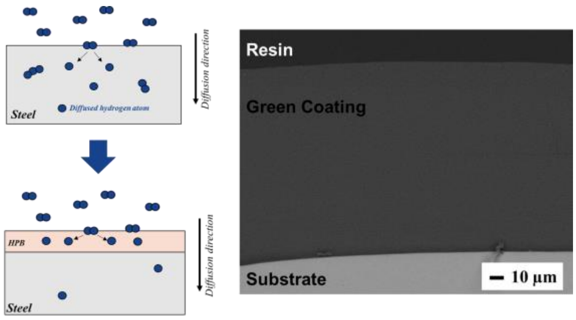


Figure 1: Figure illustrating the schematic principle of an HPB (left), SEM image of the green alumina coating fabricated by the electrophoretic process (right).

References:

1. Laadel, N. E., El Mansori, M., Kang, N., Marlin, S., & Boussant-Roux, Y. (2022). Permeation barriers for hydrogen embrittlement prevention in metals–a review on mechanisms, materials suitability and efficiency. International Journal of Hydrogen Energy, 47(76), 32707-32731.
2. Besra, L., & Liu, M. (2007). A review on fundamentals and applications of electrophoretic deposition (EPD). Progress in materials science, 52(1), 1-61.
3. Nemanič, V. (2019). Hydrogen permeation barriers: Basic requirements, materials selection, deposition methods, and quality evaluation. Nuclear Materials and Energy, 19, 451-457.

Reactive sputtering of ceramic hydrogen barrier coatings

A. Zirbel [1,2,*], L. Haus [1], S. Hübner [4], M. Müller [1], J. Riedel [3], S. Ulrich [2]
[1] Robert Bosch Manufacturing Solutions GmbH, Stuttgart, Germany
[2] Institute for Applied Materials, Karlsruher Institute ofTechnology, Karlsruhe, Germany
[3] iChem Analytics GmbH, Detmold, Germany
[4] Singulus Technologies AG, Kahl am Main, Germany

Abstract:

In the course of the energy transition and the establishment of renewable energies, hydrogen as an energy carrier plays an important role both as a fuel in direct combustion and in energy conversion in fuel cells and electrolysers (figure 1). However, new challenges arise through its properties. As the smallest and lightest element, hydrogen requires only a very low activation energy for diffusion in solids, even at low temperatures. This becomes very clear under consideration that the diffusion of hydrogen in iron at room temperature is about 10 to 15 orders of magnitude higher than that of carbon, nitrogen or oxygen. The high mobility and interaction of the penetrating hydrogen with the lattice of some metals results in a high risk of hydrogen embrittlement. Affected components should therefore be protected by a thin barrier layer. Important for the effectiveness of such a hydrogen barrier layer is a high layer quality, hence methods of physical vapor deposition (PVD) are particularly suitable. In this work, reactive magnetron sputtering was chosen as a deposition method in both batch and inline processes. Promising coating systems were initially developed in industrial batch coaters. These systems have been used for decades for the mass production of wear protective coatings on rotationally symmetrical components of the combustion engine power train of motor vehicles. For coating large-area components of electrochemical energy converters, the switch from batch coaters to inline processes is economically necessary. Therefore, the most promising layer systems – in our case tungsten carbide, zirconium nitride, diamond-like carbon - were transferred from batch to inline technology. An electrochemical and a gas-driven hydrogen permeation measurement method were used to evaluate the barrier effect. The results of both methods are in good agreement. With a permeation reduction factor (PRF) in the order of up to 10^4 and a long-term stability of the barrier effect at room temperature of over 68h, excellent hydrogen permeation barrier layers could be confirmed.

Keywords: energy transition, hydrogen permeation barrier coating, PVD, reactive magnetron sputtering, tungsten carbide, zirconium nitride, diamond-like carbon, permeation reduction factor

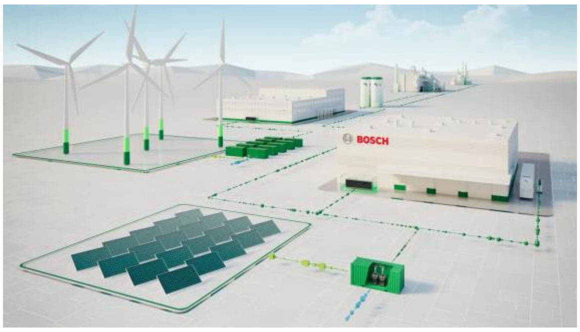


Figure 1: Industrial green hydrogen production plant.

References:

1. Mehrer, H. (2007) Diffusion in Solids: Fundamentals, Methods, Materials, Diffusion-Controlled Processes, *Springer-Verlag*, 313-326.
2. Nemanič, V. (2019) Hydrogen permeation barriers: Basic requirements, materials selection, deposition methods, and quality evaluation, *Nuclear Materials and Energy*, 19, 451-457.

Influence of operating parameters of nanosecond pulsed dielectric discharge on the CH_4 reforming with CO_2

TEJA VARDHAN REDDY THURPU [1,2,*], Elodie FOURRE [1], Sylvie ROSSIGNOL [2], Catherine BATIOT DUPEYRAT [1]
[1] Institut de Chime des Milieux et Matériaux de Poitiers,IC2MP,UMR-7285,France.
[2] Institut de recherche sur les céramiques (IRCER) UMR 7315, Limoges, France.

Abstract:

Non-thermal plasma (NTP) has been providing an innovative and attractive solution for CH_4 reforming with CO_2 into clean fuels and valuable chemicals, at ambient pressure and low temperature [1]. In NTPs, dielectric barrier discharge (DBD) attracted significant attention due to its flexible design and easy operation under mild conditions. Conventional DBDs with AC power can lead to overheating and reactor damage [2]. Nanosecond pulsed DBD plasma is gaining interest in converting CH_4 and CO_2 as it shows enhancing energy efficiency of the plasma chemical process by generating high instantaneous power and electric fields. This study investigated CH_4 reforming with CO_2 using nanosecond pulsed DBD plasma. The effects of pulse parameters (pulse repetition frequency and pulse width), discharge power, and molar ratios were evaluated on the reaction performances. All the experiments were conducted at a constant flow rate of 40 mL/min and molar ratio (CH_4/CO_2; 1/1). At constant power (16 W) the increase in pulse width from 150 ns to 175 ns enhanced CH_4 conversion from 18.6 % to 21.1 %, and CO_2 conversion almost similar around 8.0 % while the products' selectivity remained constant. Note that a temperature increase from room temperature to 200 °C was observed whatever the pulse width.

CH_4 conversion was also enhanced by increasing the pulse frequency (Fig 1a), while the deposited power was fixed at 16 W. The best CO_2 and CH_4 conversions were 8.0 % and 21.1 %, respectively, at 10 kHz.The selectivity to CO decreased by increasing the pulse frequency, while the selectivity to C_3H_8 and methanol was increased. The selectivity to other products (C_2 hydrocarbons) remained constant at different pulse frequencies (Table 1). The best hydrocarbon selectivities were obtained for C_2H_6 (21.3 %), followed by C_3H_8 (8.1 %). Methanol was identified with a low selectivity of 1.8 %. Increasing the deposited power from 10 to 16 W leads to an increase in both CH_4 and CO_2 conversion as expected (Fig.1b).

Keywords: Nanosecond pulse, DBD, dry reforming

The maximum selectivities of CO and C_2 hydrocarbons were obtained at the lowest discharge power while C_3H_8 and methanol were obtained at high discharge power (Table 2). Adding oxygen (10%) in the feed gas at a constant flow rate of 40 mL/min led to a shift in the selectivity of products from hydrocarbons to CO. A CO selectivity close to 70 % was reached for a CH_4 conversion of 26.1 % at a discharge power of 15 W. It shows that a pulse plasma system is effective for selective synthesis of carbon monoxide, an important intermediate product for the chemistry sector.

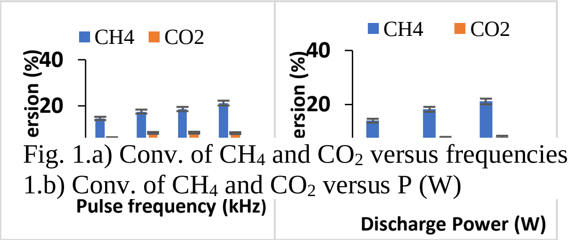


Fig. 1.a) Conv. of CH_4 and CO_2 versus frequencies
1.b) Conv. of CH_4 and CO_2 versus P (W)

Table 1 Selectivities of products at different frequencies

F (kHz)	Selectivity (%)					C.B (%)
	C_2H_6	C_3H_8	CO	C_2	CH_3OH	
4	21.4	6.5	56.0	1.6	1.4	86.9
6	20.9	7.0	52.0	1.6	1.5	83.0
8	21.2	7.0	48.6	1.6	1.5	79.9
10	21.3	8.1	48.7	1.7	1.8	81.6

Table 2: Selectivities of products at different powers

Power (W)	Selectivity (%)					C.B. (%)
	C_2H_6	C_3H_8	CO	C_2	CH_3OH	
10	22.5	7.1	56.6	2.0	1.3	89.5
13	21.3	7.8	52.2	1.8	1.6	84.7
16	21.3	8.1	48.7	1.7	1.8	81.6

References:

1. Mei, Danhua, et al. "CH4 reforming with CO2 using a nanosecond pulsed dielectric barrier discharge plasma." *Journal of CO2 Utilization* 62 (2022): 102073.
2. Wang, Xiaoling, et al. "Nanosecond pulsed plasma-assisted dry reforming of CH4: The effect of plasma operating parameters." *Applied energy* 243 (2019): 132-144.

Original liquid DBD plasma reactor coupled with membrane filtration

N. Mougel, E. Fourré, B. Teychené
IC2MP, Institute of Chemistry of Media and Materials of Poitiers, University of Poitiers, France

Abstract:

The alarming increase of pollutants in water sources, such as endocrine-disrupting chemicals, pharmaceuticals and microorganisms, poses a serious threat to human health, aquatic life, and the environment. The development of efficient water treatment technologies for both drinking water and wastewater reuse has become a global priority in scientific research. Advanced oxidation processes (AOPs) utilizes powerful oxidants to break down pollutants into harmless molecules, the most widely used and proven system beeing the coupling of H_2O_2 with O_3 (1). Ozone is typically produced in gaseous form through corona discharge across a dielectric. The high cost of gas-liquid contactors and potential VOC stripping pushed research to develop more advanced processes. Membrane contactors show promises to overcome these limitations. Indeed, gas-liquid transfer through hydrophobic membranes is governed by diffusion, allowing for better control of mass transfer. Recently, AOP induced by non-thermal plasma (NTP) processes have been developed (2). Current research is focused on the study of dielectric barrier discharge (DBD) reactors on a falling liquid film. These falling film reactors show very good electrical efficiency and degradation performance due to a large interfacial surface between the plasma and the liquid. The approach presented here offers the opportunity to intensify this process by introducing a membrane separation that allows both the separation of the components and the production of a falling liquid film (Figure 1). The plasma-membrane reactor, in terms of active species transfer from the gas phase to the liquid phase, notably H_2O_2 and O_3 was validated. An acidification of the media was observed, due to diffusion of NO_x in the liquid phase. H_2O_2 and O_3 concentrations were found independent of gas flow rate but depended on liquid residence time (~ limitation of liquid mass transfer). Finally, two pollutants, carbamazepine (CBZ) and desethylatrazine (DEA) were efficiently degraded, up to 75 % (Figure 2).

Keywords: AOP - membrane filtration - non-thermal plasma - liquid falling film - pollutant removal - ozone

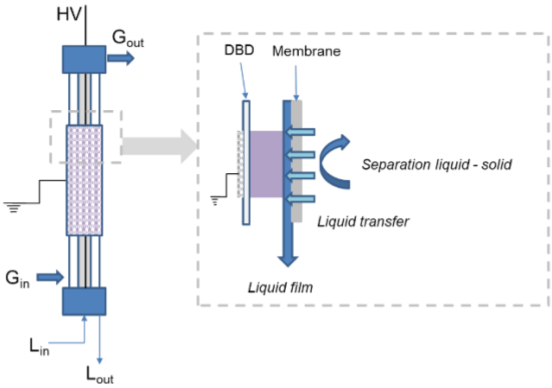


Figure 1: Scheme of membrane- plasma reactor

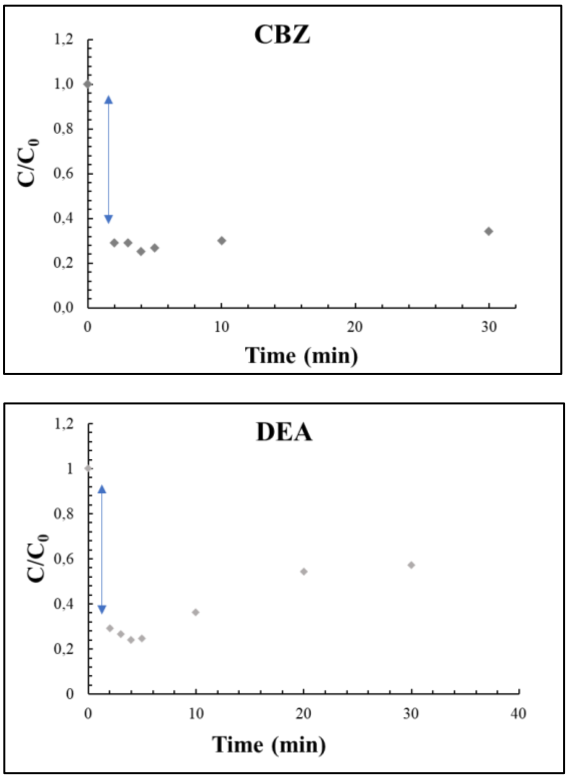


Figure 2: kinetic of degradation of CBZ and DEA as a function of the plasma treatment time

References:

1. K.Wei *et al.*, Membrane separation coupled with electrochemical advanced oxidation processes for organic wasrtewater: a short review, Membrane (2020) 10, 337
2. E.Mouele *et al.*, A critical review on ozone and co-speices, generation and reaction mechanisms in plasma induced by dielectric barrier discharge technologies for wastewater remediation, J. Env. Chem. Eng. (2021), 9, 105758

Hydrogen Production from NH_3 in a Ferroelectric Packed-Bed Plasma Reactor

M. Ruiz-Martín [1,2], S. Marín-Meana[1,2], A. Megías-Sánchez[1,2], M. Oliva-Ramírez[1,2], J. Cotrino[1,2]
A.R.González-Elipe[1], A. Gómez-Ramírez[1,2]

[1] Laboratory of Nanotechnology on Surfaces and Plasma, Instituto de Ciencia de Materiales de Sevilla (CSIC-Universidad de Sevilla), Seville, Spain
[2] Departamento de Física Atómica, Molecular y Nuclear, Universidad de Sevilla, Seville, Spain

Abstract:

Plasma decomposition reactions are used for various gas phase chemical processes including the decomposition of ammonia to generate hydrogen [1,2,3,4]. As an ammonia distribution grid already exists, NH_3 is considered to be the cost-effective carrier enabling in-situ recovery of hydrogen. In this work we show that pure ammonia can be effectively decomposed at atmospheric pressure and ambient temperature using a packed-bed plasma reactor moderated with $BaTiO_3$ ferroelectric pellets without catalyst (see Figures 1 and 2). The decomposition rate and energy efficiency of this ferroelectric barrier discharge reactor have been monitored as a function of applied voltage (up to a maximum value of 2.5 kV) and flow rate. For each operating condition reaction efficiencies have been correlated with the parameters defining the electrical response of the reactor. It is found that plasma current and volume inside the reactor and hence the energy efficiency of the process and the decomposition rate vary with the applied voltage and the flow of ammonia (a maximum decomposition rate of 14% and an energy efficiency of 150 LH_2/kWh has been determined under optimized operation conditions). The role of back reactions (i.e. $N_2 + 3H_2 \rightarrow 2NH_3$) in decreasing reactor performance is another key effect affecting the overall efficiency for the ammonia decomposition. The possibilities of ferroelectric barrier discharge reactors to induce the decomposition of ammonia and the importance of keeping the operating temperature below the Curie temperature of the ferroelectric material are highlighted.

Keywords: hydrogen production, ammonia decomposition, plasma, atmospheric pressure, packed-bed plasma reactor, microdischarges, barium titanate $BaTiO_3$.

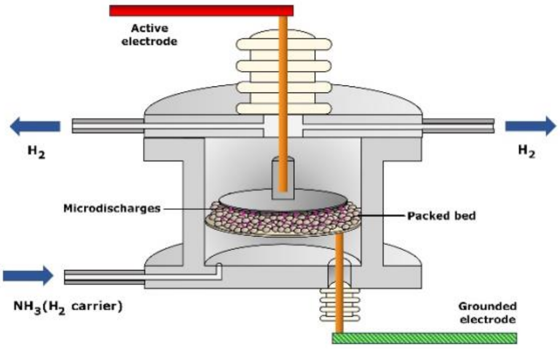


Figure 1: Sketch of the packed-bed DBD plasma reactor.

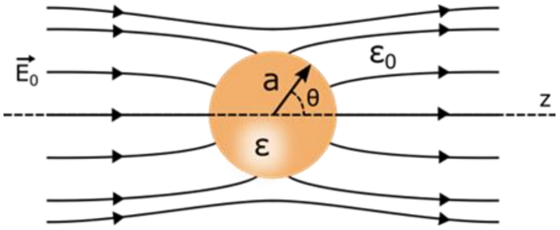


Figure 2: Distortion of the electric field in the proximity of a pellet with a dielectric constant ϵ and radius a. The moderator material induces an increase in the electric field and a higher accumulation of surface charge. These effects contribute to facilitating specific reaction mechanisms and enhancing the efficiency of certain chemical processes.

Acknowledgements:

The authors acknowledge projects PID2020-114270RA-I00, and PID2020-112620GB-I00 funded by MCIN/AEI/10.13039/501100011033, project TED2021-130124A-I00 funded by AEI/10.13039/501100011033/Unión Europea Next Generation EU/PRTR and projects P18-RT-3480, US-1381045, and US-1380977 funded by Conserjería de Economía, Conocimiento, Empresas y Universidad de la Junta de Andalucía (PAIDI-2020) and Programa Operativo Feder 2014-2020. M. Oliva-Ramírez acknowledges financial support from Grant IJC2020-045087-I funded by: MCIN/ AEI

/10.13039/501100011033 and the European Union NextGeneration EU /PRTR and S. Marín Meana acknowledges fnancial support from Grant PRE2021- 100465 funded by MCIN/AEI/10.13039/501100011033 and FSE+

References:

1. Ruiz-Martín, M., Marín-Meana, S., Megías-Sánchez, A., Oliva-Ramírez, M., Cotrino, J., González-Elipe, A. R., Gómez-Ramírez, A. (2023). H_2 production from NH_3 in a $BaTiO_3$ moderated ferroelectric packed-bed plasma reactor. *Plasma Chemistry and Plasma Processing*, *43*(6), 2093–2110.
2. Andersen, J. A., Christensen, J. M., Østberg, M., Bogaerts, A., Jensen, A. D. (2022). Plasma-catalytic ammonia decomposition using a packed-bed dielectric barrier discharge reactor. *International Journal of Hydrogen Energy*, *47*(75), 32081–32091.
3. Wang, L., Zhao, Y., Liu, C., Gong, W., Guo, H. (2013b). Plasma driven ammonia decomposition on a Fe-catalyst: Eliminating surface nitrogen poisoning. *Chemical Communications*, *49*(36), 3787.
4. Yi, Y., Wang, L., Guo, Y., Sun, S., Guo, H. (2018). Plasma-assisted ammonia decomposition over Fe–Ni alloy catalysts for COx-free hydrogen. *AIChE Journal*, *65*(2), 691–701.

Posters Abstracts

Design and synthesis of advanced nanostructured coatings with hydrophobic/ icephobic properties for aviation

Z. Sideratou[1], D. Tsiourvas[1], F. Katsaros[1], K. Spyrou[1,2], A. Sapalidis[1], S. Papageorgiou[1], P. Booker[3]
[1]Institute of Nanoscience and Nanotechnology, NCSR “Demokritos”, Aghia Paraskevi Attikis, Athens 15310, Greece
[2] Department of Materials Science and Engineering, University of Ioannina, Ioannina 45110, Greece
[3]bionanovate Ltd., Poole, BH17 7BP, UK

Abstract:

The efficiency of modern transportation is severely compromised by the prevalence of turbulent drag and icing. On the other hand, ice accretion on the airframe affects the performance by reducing lift and increasing weight and drag, affecting aircraft aerodynamic performance, stability and controllability, causing a series of aircraft flight accidents.

The current study is focused on the development of hydrophobic/ice-phobic nanostructured top coatings, which can not only exhibit improved aerodynamic efficiency but at the same time they prevent icing on the aircraft[1]. The main objective was the preparation of functionalized nanoparticles bearing fluoroalkyl groups in order to induce hydrophobic properties resulting in reduction of ice adhesion and increase UV resistance.

To this end organic–inorganic hybrid silica nanoparticles were prepared through a biomimetic silicification process at ambient conditions in the presence of amino terminated dendritic polymer with fluorinated silica precursors. The developed silica nanoparticles bearing fluoroalkyl groups in their surface exhibited platelet like morphology with dimension ~ 25-30 nm and thickness of 2-4 nm. In addition, carbon-based nanostructures (CNTs and GO) were also used as nanofillers. Regarding CNTs, both covalent (chemical) and non-covalent (physical) modifications were employed in order to attach fluoro- containing functional polymers. In the case of GO, exfoliated structures were developed using amino terminated dendritic polymers of an appropriate molecular weight[2] and subsequently the hybrid polymer/GO materials were further modified with fluoroalkyl chains through covalent bonding.

The developed nanoparticles were thoroughly characterised by a variety of techniques including SEM, TEM, AFM, FTIR and used as nanofillers in commercially available silicon-based emulsions for the development of top-coats that can be applied on aircraft. The developed coatings exhibited increased water contact angles and reduced surface roughness. Finally, the performance of the novel nanostructured coatings was evaluated by ice adhesion experiments, which releveled a significant reduction in ice pull-off force (~ one order of magnitude) in regards to the uncoated aircraft paints.

Keywords: Silica nanoparticles, CNTs, GO, hydrophobic top-coats, icephobic surfaces, aviation coatings.

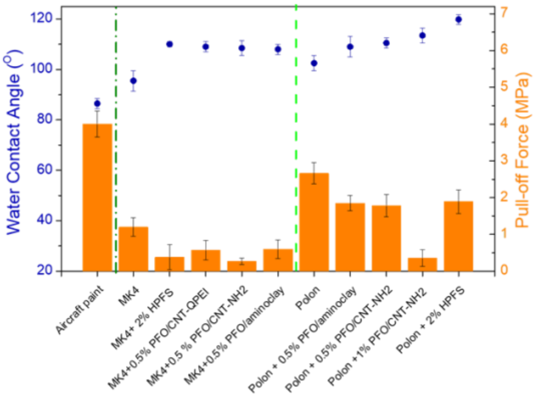


Figure 1: Water CA and pull-off force for the developed nanostructured top-coats in comparison to original aircraft paint

References:

1. Golovin, K., Kobaku, S.P.R., Lee, D.H., DiLoreto, E.T., Mabry, J.M., Tuteja, A. (2016) Designing durable icephobic surfaces, Sci. Adv., 2, e1501496
2. Tsoufis, T., Katsaros, F., Sideratou, Z., Kooi, B.J., Karakassides, M.A., Siozios, A. (2014) Intercalation Study of Low-Molecular-Weight Hyperbranched Polyethyleneimine into Graphite Oxide, Chem. Eur. J. 20(26), 8129-8137

Acknowledgements:

This work was partially funded by EU under the FP7 PEOPLE-2012-IAPP-SANAD project (Grant Agreement No 324443) and the NCSR Demokritos internal project EE-11968, entitled “Synthesis and characterization of nanostructured materials for environmental applications”.

Methodological study of the deposition parameters of aminopropyl silsesquioxane on the surface of glass vials using aqueous solution

T. Pastore [1,2,*], G. Trevisi [1,] F. Casoli [1] , L. Savio [3], M. Di Maro [4], G. Gautier [4], M. G. Faga [4],
D. Costa [5], M. Poncini [5], D. Faverzani [5]
[1] IMEM-CNR Institute , Parma, Italy; [2] University of Parma, Parma, Italy; [3] IMEM-CNR Institute, Genova, Italy; [4] STEMS-CNR Institute, Torino, Italy; [5] BormioliPharma spa, Parma, Italy

Abstract:
The glass packaging industry faces a significant challenge in maintaining the strength of glass during the manufacturing process, so smoothness on production and filling lines is an essential requirement. Current methods involve using metal primers and polymer lubricants, but these have durability issues. An alternative approach could be based on organosilane coupling agents, hybrid compounds capable of creating a durable interface between organic polymers and inorganic substrates such as glass, effectively used as adhesion promoters[1]. Research primarily focuses on their use in organic or alcoholic solutions, usually in an acidic environment, to promote hydrolysis and generate silanols capable of binding inorganic substrates. It is worth noting that water-based solutions are preferable from the point of view of implementing industrial processes[2]. In this work we study the deposition of aminopropylsilsesquioxane on the external surface of glass vials via aqueous solution considering different deposition parameters: water rinsing, silane concentration and immersion time. Plasma cleaning precedes the process to expose OH groups on the glass surface, as confirmed by contact angle observations. Characterization via AFM [Fig.1] and SEM microscopies reveals increased surface roughness due to the coating, with a correlation between coating thickness and silane concentration established. FT-IR and XPS analyses identify functional groups in the coatings, highlighting the presence of desirable free NH_2 amine groups, suitable for subsequent functionalization with organic lubricants. Tribological measurements were performed on a selected set of coated glass samples with and without the lubricant applied. The results showed that the presence of the coating did not alter the friction coefficient of the lubricant. However, the increase in mechanical stress leads to a sudden increase in the coefficient, indicating the detachment of the coating.The results of this study show the possibility of developing an innovative glass coating approach based on water-based solutions of non-toxic organosilane compounds, opening the way to eco-sustainable industrial processes of great interest for the glass packaging sector.

Keywords: organosilane, adhesion promoters, water-based coatings, glass surface modifications, deposition parameters.

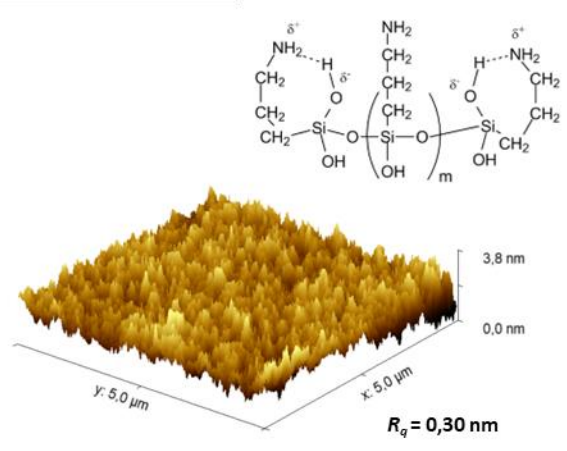


Figure 1: Molecular structure of the organosilane compound used and corresponding AFM image of the coated glass surface.

References:

1. Glosz, K. *et al.* (2020), Siloxanes—versatile materials for surface functionalisation and graft copolymers, *Int. J. Mol. Sci.* 21, 6387.
2. Spirk, S. *et al.*(2010),Surface modifications using a water-stable silanetriol in neutral aqueous media, *ACS Appl. Mater. Interfaces*, 2, 2956.

Multilayered CVD coated tools with texturized Al_2O_3 layer for machining of high strength special steels

A. Pinto [1], J. Ferreira [1], F.J. Oliveira [1,*], A.V. Girão [1], C.M. Fernandes [2], D. Figueiredo [2]
[1] CICECO & Department of Materials and Ceramic Engineering, University of Aveiro, 3810-193 Aveiro, Portugal
[2] Palbit S.A., Palhal - Rua das Tílias, 3850-582, Branca, Albergaria-a-Velha, Portugal

Abstract:

In the current era of energy transition, the manufacturing of new electric vehicles changed, as lighter and stronger materials such as high strength special steels are needed. Thus, harder and stronger tools to machine such materials are also required.

The main objective of this work is further improvement of cutting tools with multilayer chemical vapor deposition (CVD) coatings of TiN-TiCN-Al_2O_3-TiN for turning and milling operations, focusing on innovation of the textured Al_2O_3 layer or the final deposited layer. The influence of the thickness of each layer and total thickness of the multilayered coating on the machining performance of high strength special steels was evaluated. Turning and milling operations were also carried out to test the tools with an additional coating layer of AlCrN deposited by high power impulse magnetron sputtering (HIPIMS) on top of the final TiN layer.

Identification of the different crystalline phases and texture assessment was carried out by X-rays Diffraction (XRD). Scanning Electron Microscopy with Energy Dispersive Spectroscopy (SEM-EDS) enabled morphological characterization and mapping of the elemental distribution within the multilayered coatings. The effect of each layer on the final performance of the cutting tool was assessed by Vickers microhardness testing.

Machining performance in continuous turning demonstrated that the tool life of the texturized insert is approximately twice as long as the non-texturized one. The latter is most probably due to the combination between the slight increase in the hardness of the texturized alumina layer and its microstructure. High thickness texturized Al_2O_3 layer thickness did not show any brittle failure and increased thickness demonstrated a relevant positive impact. Finishing the multilayered tools with a coating layer of AlCrN also proved its benefits in the cutting and turning of high strength special steels. In the near future, this work foresees tools with universal application and superior productivity conditions.

Keywords: CVD, multilayer, textured alumina, tools, HIPIMS, wear

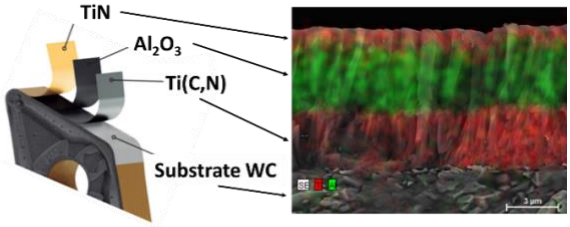


Figure 1: Figure illustrating the studied multilayered CVD coating (TiN-TiCN-Al_2O_3-TiN) of the cutting tools used in machining of high strength special steels.

References:

1. Micallef, C., Zhuk, Y., Aria, A. I. (2020) Recent progress in precision machining and surface finishing of tungsten carbide hard composite coatings, *Coatings*, 10(8), 731.
2. Bobzin, K. (2017) High-performance coatings for cutting tools, *CIRP J. Manuf. Sci. Technol.*, 18, 1–9.
3. M'Saoubi, R., Alm, O., Andersson, J. M., Engström, H., Larsson, T., Johansson-Jöesaar, M. P., Schwind, M. (2017) Microstructure and wear mechanisms of texture-controlled CVD α-Al_2O_3 coatings, *Wear*, 376–377, 1766–1778.

Acknowlegments:

This study was funded by the PRR – Plano de Recuperação e Resiliência and by the NextGenerationEU funds at Universidade de Aveiro, through the scope of the Mobilizing Project of the Innovation Green Agendas "Hi-rEV – Recuperação do Setor de Componentes Automóveis" (Project nº 64 with the application number C644864375-00000002)

Comparative investigation of the biodegradable control properties of post- and simply PEO coated Mg alloy

Zeeshan Ur Rehman and Bon Heun Koo*
College of Mechatronic Engineering, Changwon National University, Changwon, Gyeongsangnam–do, 51140, Republic of Korea
zeeshan.physics@gmail.com, bhkoo@changwon.ac.kr

Abstract

Due to the optimum mechanical, excellent biocompatibility characteristics of Magnesium and its alloys, they are considered as a potential solution for various orthopedic implants and devices. However, the wise spread realization of this concept is encountered by the high degradation rate of Mg in human body fluid/Chloride containing environment and consequently the sequential deterioration of it mechanical properties. This would make the implant failure possibility very high, before the bone tissues recovery, thus a sustainable surface treatment is mandatory before promoting the widespread use of Mg in biomedical implants.

In this study, a detailed analysis of the simply coated Mg samples using plasma electrolytic oxidation techniques and Post PEO coated samples were carried out. The finally obtained samples were characterized using SEM, EDS, FTIR, for surface molecular and microstructural observations. Furthermore, biodegradation properties were analyzed using EIS, Potentiodynamic polarization and weight loss method to study the degradation rate of the coated samples. The result showed that post-anodization treatment under optimized concentration was advantageous in controlling the degradation of the alloy.

Keywords: AZ31, Orthopedic implant, Post PEO, biodegradation, SEM

The high-temperature stability and mechanical properties of multicomponent TiTaZrHf$_x$N coatings prepared by reactive DC magnetron sputtering and High target utilization sputtering

L. Kvetková [1], P. Hviščová [1] , F. Lofaj [1] , M. Mikula [2,3] T. Roch [2]
[1] Institute of Materials Research of the Slovak Academy of Sciences, Watsonova 47, 040 01 Košice, Slovakia
[2] Department of Experimental Physics, Faculty of Mathematics, Physics and Informatics, Comenius University in Bratislava, Mlynska Dolina F2, Bratislava 842 48, Slovakia
3 Institute of Materials and Machine Mechanics, Slovak Academy of Sciences, Dúbravská cesta 9, Bratislava 845 11, Slovakia

Abstract:

Currently, considerable efforts are devoted to the research of multicomponent coatings which are expected to provide enhanced hardness, wear resistance, adhesion, thermal and electrical conductivity, chemical resistance to aggressive substances, and low density. In comparison with two- and three-component systems, an increase in mechanical properties was observed for multicomponent coatings. An increase in their hardness to 36.9 GPa and a slight dependence of their structure and mechanical properties on the variation of deposition parameters - reactive gas flow rate, substrate bias and substrate temperature – were demonstrated. Kirnbauer et al. studied the mechanical properties and thermal stability of Hf-Ta-Ti-V-Zr-N coatings prepared by reactive magnetron sputtering; they were varying the nitrogen flow rate in the range of 30 - to 45 %. The highest hardness of 32.5 ± 0.8 GPa was obtained at fN_2= 45%, (where fN_2= N_2/ (Ar + N_2)). In this study, we report the influence of nitrogen inflow, fN_2 0 to 8 %. Main bjectives of the study are focused on understanding the structure evolution mechanisms during growth and on analyzing the relationships between structure, phase composition and properties of multicomponent coatings by optimizing the magnetron sputtering processes. Chemical complexity of multicomponent coatings and the absence of sintering additives can strongly influence the mechanical, thermal and diffusion behavior, the research of which is currently very topical. The The variability of the components will allow to study the thermal stability of nitride-based multicomponent coatings.

Keywords: multicomponent coatings, DC magnetron sputtering, high target utilization sputtering, thermal stability, structure, phase composition, mechanical properties.

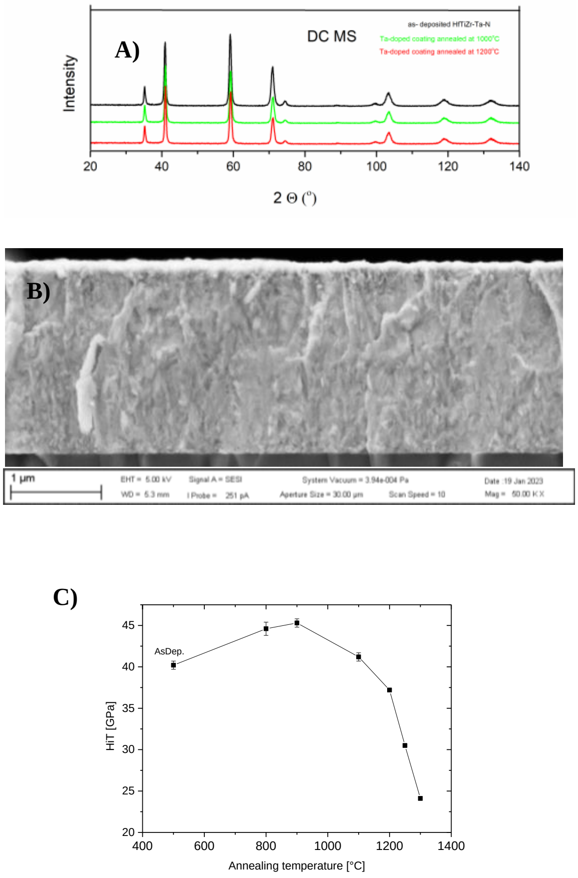


Figure 1: Figure illustrating A) the XRD results of annealed coating as deposited, annealed at 100 and 1200 °C, B) SEM/FIB annealed coating, C) nitrogen inflow ependence on hardness.

References:

1. Y Zhang a kol, a kol. Prog. Mater. Sci. 61 1 (2014).
2. Pogrebnjak a kol, Matt. Lett. (2018)
3. A. Kirnbauer a kol., Surf. Coatings Tech. 389 (2020)
4. F. Lofaj a kol, J. Eur. Ceram. Soc., 40 (2019)

Nanoarchitectures with Cu-Ag embedded clusters for enhanced localized surface plasmon resonance detection

Sanjay Kumar[1], Hana Maskova[1,2], Lenka Doudová[1,2], Pavel Pleskunov[3], Anna Kuzminova[3], Pavel Curda[1], Libor Hejduk[1,2], Jan Sterba[1], Ondřej Kylián[3], Ryan O. M. Rego[1,2], Vitezslav Stranak[1*]
[1]University of South Bohemia, Faculty of Science, Branisovska 1760, 37005, Ceske Budejovice, Czech Republic
[2]Biology Centre ASCR, Institute of Parasitology Branisovska 31, 37005 Ceske Budejovice, Czech Republic
[3]Charles University, Faculty of Mathematics and Physics, V Holešovičkách 2, 180 00, Prague 8, Czech Republic

Abstract:

The study investigates the enhancement of localized surface plasmon resonance (LSPR) response utilized for sensing applications. The concept is based on a nanocomposite platform of C:H:N:O matrix with anchored Ag and Cu nanoparticles, approximately 25 nm in diameter. The C:H:N:O plasma polymer film was deposited by magnetron sputtering of nylon 6.6 target while the nanoparticles were produced in a gas aggregation system. The nanoparticles, made of plasmonic metals, exhibit absorption in the visible wavelength region and spectral absorption shift caused by optical and electrical changes near the nanoparticles. transducer surface. Tailored design and nanoarchitecture of Ag – Cu nanoparticles were studied with the aim of improving the LSPR response, i.e. the sensitivity, of the transducer platform. The study of the dimer-like and tetramer-like architecture of Ag-Cu nanoparticles. We have experimentally demonstrated and confirmed through simulations that the near-field interactions of Cu and Ag nanoparticles significantly enhance plasmonic absorption. The LSPR plasmonic shift was observed to be enhanced by more than half an order of magnitude if the nanoparticle arrangement is defined properly.

The developed platform was tested for detecting specific anti-DbpA antibodies in real-life negative and positive sera samples. Distinguishable differences between the negative and positive sera of LSPR response, coupled with high sensitivity, selectivity, and stability of the nanocomposite, suggest potential for rapid, low-cost, point-of-care testing

Acknowledgment: The work was financially supported by the Czech Science Foundation Agency through the project 21-05030K.

Keywords: localized surface plasmon resonance, plasmonic nanoparticles (Ag, Cu), nylon plasma polymer films (C:H:N:O), nanocomposites, transducer, Lyme disease antibodies, enhanced detection

Tribogical properties of DLC deposited by PE-CVD under high Vacuum

Bo Ram Cho [1],Tae-Hwan Jang [2],Tae-Gyu Kim [2],Inkang Heo [1],Bokseong Choe [1],Jong Hyoung Kim [1*]
[1] High-Tech Bearing Technology center, Korea Institute of Industrial Technology, Yeongju, Korea
[2] Department of Nanomechatronics Engineering, Pusan national university, Busan, Korea

Abstract:

Recently, research on tribology characterstic of aerospace machines has increased. In particular, friction of bearings plays an important role in the motion of a rotating body in aerospace machine. Normally, use of the lubricant or grease in bearing system is strongly needed to decrease frition and wear, but it is restricted in a vacuum environment. Therefore, this study is focued on understanding of tribology charaterteristc in a dry environment.

This study compared friction characteristics with disks made of general SUJ2 materials by manufacturing DLC (Diamond like carbon) coated disks with a plasma-reinforced chemical vapor deposition (PE-CVD) process applicable to substrates.

To confirm this, friction properties were compared and tested using the Pin-on-Disk method using the Precision Rotary Vacuum Tribometer (TE91) of Phoenix Tribology Ltd. The composition of the test equipment has a jig that fixes the disk and Pin, respectively, and the load is controlled by pneumatic pressure and a load is applied in the direction of Pin from the disk.

In vacuum environment conditions, the load was fixed at 20 N, three pins were rotated, and the coefficient of friction was checked during friction with the disk. After the friction wear test was completed, the friction surface was analyzed using Fe-sem, and the wear depth and wear amount were analyzed before and after the test through a three-dimensional wear measurement device.

This experiment compared and analyzed the friction coefficient and wear amount of SUJ2 and DLC coated disks, and will be used as basic data for fatigue contact durability tests of DLC coateing in a vacuum environment in the future.

Keywords: vaccum, friction characteristics, DLC, PE-CVD, Rotary Vacuum Tribomter,Pin-on-disk

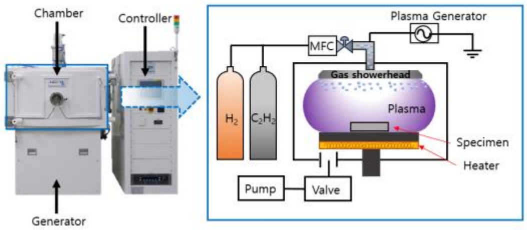


Figure 1. PE-CVD process schematic diagram

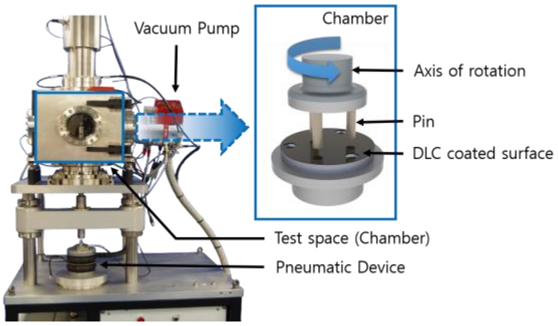


Figure 2. Experiment equipment(Rotary Vacuum Tribomter) & Pin-on-Disk test concept diagram

References:

1. Shi Jiadong, Ma Guozheng, (2022) The tribological performance in vaccum of DLC coating treated with graphene spraying top lalyer, *Diamond & Related Materials*, 125, 108998-109005
2. Bernd Rubig, Daniel Heim, (2017) Tribological behavior of thick DLC coating under lubricated conditions, *Surface & Coating Technology*, 314, 13-17

Structural and mechanical properties of laminate-type thin film SWCNT/ SiO_XN_Y composites

E. Shmagina[1,*], O. Volobujeva[1], M. Antonov[2], S. Bereznev[1]

[1] Department of Materials and Environmental Technology, Tallinn University of Technology, Tallinn, Estonia

[2] Department of Mechanical and Industrial Engineering, Tallinn University of Technology, Tallinn, Estonia

Abstract:

Environmental factors is critically affecting performance and lifetime of solar cells. The high weight, brittleness, and inflexibility significantly limit the application of standard glass protection. Hence, the selection of optimal protection is promising both from a technical and economic point of view. Development of a nanocomposite films in a matrix/filler configuration is a proven strategy for modifying and tailoring the unique properties of the final material based on the requirements for its application [1].

We report on the technology for producing thin laminate-type nanocomposite coatings with horizontally oriented SWCNTs embedded as the filler in the SiO_XN_Y matrix derived from pre-ceramic 20% perhydropolysilazane (PHPS) solution in di-butyl ether. The matrix film is deposited by spin-coating (2000 rpm/60 sec) onto Mo/glass substrates, the filler film consisting of SWCNTs is introduced by simple dry transfer method [2]. Curing is carried out by low-temperature methods (thermal at 180° C/60 min or under the irradiation of UV lamps 185 and 254 nm/40 min), which allows the formation of coatings on thermo-sensitive materials.

In this work, we study the mechanical and adhesive properties of matrix films and composites based on them, as well as the connection of these properties with the structure and morphology of the samples. According to results of FTIR and SEM EDX measurenets the curing method affects the degree of transformation of the PHPS into SiO_XN_Y, as well as the cross-sectional morphology of the films. Based on the data we obtained from scratch tests with progressive loads, the curing method also strongly affects the mechanical and adhesive properties of the films. UV-cured films (Fig. 1a) exhibit greater hardness and lack of ductility, as well as very strong adhesion to the substrate after overcoming the critical load value, while thermally cured films are more susceptible to plastic deformation (Fig. 1b). This study will also permit us to discuss the long-term stability of that properties which is extremely important for the use of these composites as protective coatings.

Keywords: silicon oxynitride, single walled carbon nanotubes, protective nanocomposite coating, progressive damage test

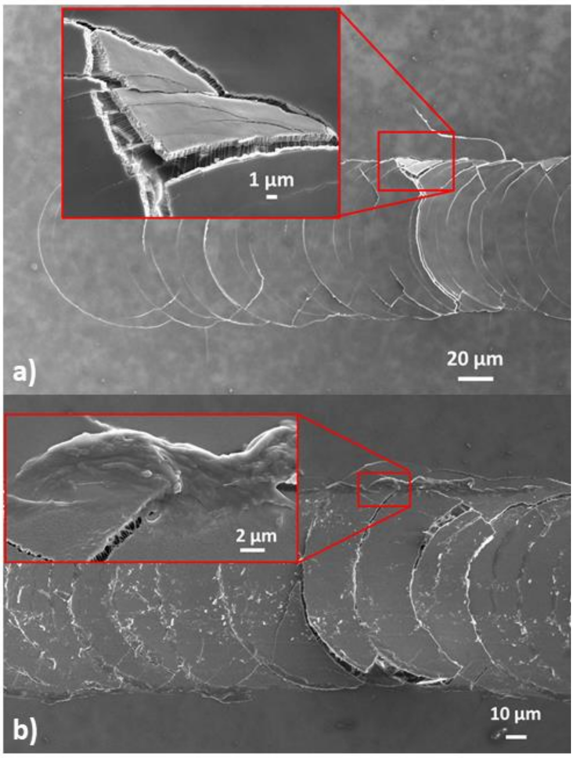


Figure 1:HR-SEM images of scratches obtained during a progressive load test on SWCNT/SiO_XN_Y composite films onto Mo/glass substrates: a) cured under UV lamps, b) thermally cured film.

References:

1. Y. Wang, G. Ding, J.Y. Mao, Y. Zhou, S.T. Han (2020) Recent advances in synthesis and application of perovskite quantum dot based composites for photonics, electronics and sensors, *Sci Technol Adv Mater.*, 21, 278–302.
2. A. Moisala, A.G. Nasibulin, D.P. Brown, H. Jiang, L. Khriachtchev, E.I. Kauppinen (2006) Single-walled carbon nanotube synthesis using ferrocene and iron pentacarbonyl in a laminar flow reactor, *Chem Eng Sci*, 61, 4393–4402.

Mechanical endurance improvement of the coated nanosurface by laser

YoungNam Jung[1], MyungYung Jeong[1 †]
Department of Opto-Mechatronics Engineering, Pusan National University ,Busan 46241, Korea

Abstract:

We proposed a hardening method of polymeric nanostructure by laser light. In the manufacture of functional nanosurface films, the use of nanoimprint technology is common. Nanoimprint technology is regarded as a low-cost and reliable patterning process. However, the formed nanopattern has low hardness due to polymer property. This property have acted as a hindrance to the commercialization of functional films. To solve this problem, many studies have been conducted in terms of materials and processes.1) When an intensively pulsed laser(IPL) is applied to a nanopatterned polymer surface, the high intensity of light enhances polymerization and improves hardness.2) In this study, we investigated the effects according to the change of IPL parameters such as voltage, pulse duration, and number of pulses. For the hardness enhancement, we proposed the optimized process condition. After polymeric nanostructured films were fabricated by nanoimprinting, each parameter of IPL was investigated independently to observe the hardness improvement. As a result of pencil hardness test according to ASTM-D3363 specification, we confirmed that there was an increase in hardness of 2H according to IPL conditions.

Keywords: Intensively Pulsed Laser. UV-nanoimprint, Polymeric hardening, Nanopatterned surface, Pencil hardness

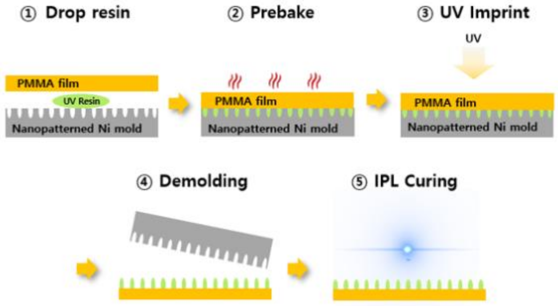


Figure 1: Overall UV-nanoimprint process with IPL process: UV-nanoimprint process followed by Intensively Pulsed Laser inducing additional cross-linking reaction in resin.

References:

1. Kim, D., Kim, D. I., & Jeong, M. Y. (2020). A Study of Mechanical Property Enhancement of Polymer Nanostructure using IPL Treatment. Journal of the Microelectronics and Packaging Society, 27(4), 113–117
2. Y. Du, B. A. Williams, L. F. Francis, and A. V. McCormick, "Pulsed irradiation for high-throughput curing applications", Progress in Organic Coatings, 104, 104 (2017).

Hybrid SLM/DED Additive Manufacturing of Ti6Al4V Alloy – Optimization of Process Parameters and Heat Treatment

A. Kocijan [1*]
[1] Institute of Metals and Technology, Ljubljana, Slovenia

Abstract:

In aerospace sector, there is a strong market need for additive manufacturing (AM) products that comprise limitless size and shape opportunities with faster building rates at relatively low cost. Ti6Al4V alloy is the most dominant titanium alloy in aerospace engineering industry due to its high strength, low density, high fracture toughness and superior corrosion properties.The idea of this study was to combine the advantages of two additive manufacturing technologies, directed energy deposition (DED) and selective laser melting (SLM), i.e. rapid manufacturing of larger parts with geometrically complex structures via a new concept called hybrid additive manufacturing approach. The main focus of the study was on optimization of process parameters of both AM techniques. The influence of different process parameters, such as laser power, scan spacing, scan strategy, scan speed, powder flow rate and layer thickness, on the microstructure, corrosion and mechanical properties had to be evaluated in order to ensure optimal printing conditions. Optimization of the boundary layer, in our case the interface between finer SLM part and rougher DED part is also crucial to achieve good bonding characteristics with appropriate powders for feedstock. In the case of longitudinal building pattern, the most optimal angle of inclinations had to be considered as well. On the other hand, heat treatment of Ti6Al4V printed parts had been considered as well. To improve the ductility, reduce thermal stresses and to achieve anticipated mechanical properties of Ti6Al4V AM manufactured products suitable heat treatments had been assessed.

An upgraded hybrid AM technology allows better customisation of products, internal structures that would be impossible to produce by conventional manufacturing methods, it also enables the building of hollow structures and therefore reduces weight of the parts, which is crucial in aerospace industry. This approach is also time and cost effective and has the potential to approach Zero Waste since used powders generated during the process can be reused.

Keywords additive manufacturing, directed energy deposition, and selective laser melting, Ti6Al4V alloy, process patameters, boundary layer, heat treatment, aerospace industry

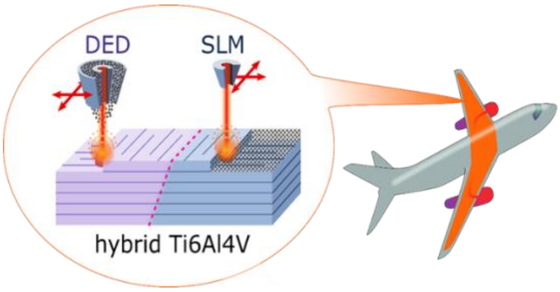


Figure 1: Figure illustrating the principal concept of hybrid additive manufacturing approach: to combine the advantages of directed energy deposition and selective laser melting for rapid manufacturing of larger parts with geometrically complex structures.

References:

1. Malej, S., Godec, M., Donik, C., Balazic, M., Zettler, R., Lienert, T., Pambaguian, L. (2023), Hybrid additive manufacturing of Ti6Al4V with powder-bed fusion and direct-energy deposition, *Mater. Sci. Eng. A*, 878, 1-17.
2. Godec, M., Malej, S., Feizpour, D., Donik, C., Balazic, M., Klobcar, D., Pambaguian, L., Conradi, M., Kocijan, A. (2021), Hybrid additive manufacturing of Inconel 718 for future space applications, *Mater. Charact.*, 172, 1-16.

Study of 2D Materials by Time-of-Flight Spectroscopy

I. Konvalina [*], E. Materna Mikmeková, L. Průcha, J. Piňos, A. Paták, M. Zouhar, I. Müllerová
Department of Electron Microscopy, Institute of Scientific Instruments of the Czech Academy of Sciences, Brno, Czech Republic

Abstract:

Graphene was discovered in 2004 and is the main representative of modern two-dimensional (2D) materials. During the last two decades, there has been a boom in 2D materials due to their unique properties and novel application potential. This increases the requirements for analytical techniques for their study and analysis.

Surface sensitive electron spectroscopy and microscopy requires theoretical and experimental studies of low energy electron transport near solid surfaces. In particular, a reliable knowledge of electron transport in the sample and across its interface is necessary to obtain quantitative information.

For our experiments we use a unique device developed in our institute. It is an ultra-high vacuum scanning low energy electron microscope (UHV SLEEM), equipped with a time-of-flight (ToF) spectrometer operating in transmission mode [1].

Graphene is lightweight and strong, and its other unique properties such as excellent electrical and thermal conductivity and transparency make it an ideal candidate for study at very low energies in the transmission mode of the electron microscope.

We performed experiments for commercial graphene samples (Ted Pella, Inc.) to obtain electron energy loss spectra (EELS). Both plasmon peaks (π and $\pi+\sigma$) are present in the measured spectra for 2 layer and 3-5 layer graphene samples (Figure 1).

To reduce the unpleasant effect of hydrocarbon contamination on the analyzed site, we used the method of in-situ cleaning by electron beam irradiation [2]. The samples were subsequently analyzed by Raman spectroscopy.

This study will analyze experimental spectra of graphene samples and other 2D materials obtained for low landing energies. We will compare the measured data with simulated momentum-resolved EELS using many-body perturbation theory, Yambo code, on top of density-functional theory, Quantum Espresso.

Keywords: graphene, 2D materials, time-of-flight spectroscopy, EELS, Raman spectroscopy, hydrocarbon contamination.

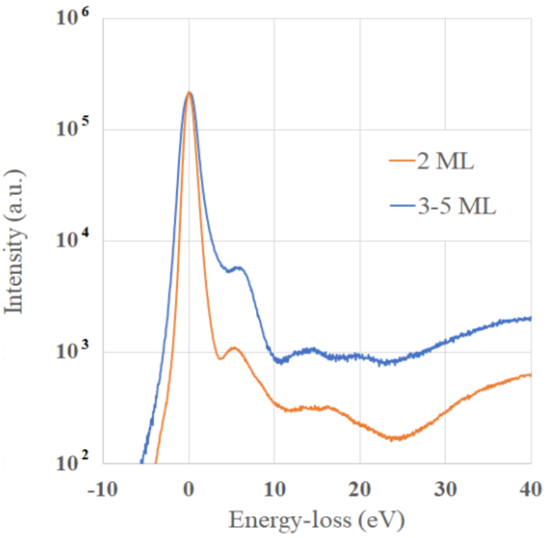


Figure 1: Experimental energy loss spectra of 2 layer and 3-5 layer graphene samples for a landing energy of 100 eV.

References:

1. Konvalina, I., Daniel, B., Zouhar, M., Paták, A., Müllerová, I., Frank, L., Piňos, J., Průcha, L., Radlička, T., Werner, W., Materna Mikmeková, E. (2021) Low-Energy Inelastic Mean Free Path of Graphene Measured by a Time-of-Flight Spectrometer, *Nanomaterials*, 11, 2435.
2. Frank, L., Mikmeková, E., Lejeune, M. (2017) Treatment of surfaces with low-energy electrons, *Applied Surface Science*, 407, 105-108.
3. This research was funded by the Technology Agency of the Czech Republic, grant number TN02000020.

Dynamics of compound droplet with dual viscosities on obstacles for center-assisted retraction

S. Yun
Department of Mechanical Engineering, Korea National University of Transportation, 27469, Republic of Korea

Abstract:

Small droplets bouncing on solid surfaces have been attracting a growing community of fundamental studies and engineering applications, such as anti-freezing of the impact surface, self-cleaning, and maintaining hygienic [1,2]. Compound materials impacting on superhydrophobic surfaces have received substantial attention because of the benefits of anti-icing and self-cleaning achieved by shorten contact time. The dynamics is in general assumed to be axisymmetric, and the contact time is still bounded below by a theoretical Rayleigh limit [1,2]. Several efforts have been made to create strategies to amend the classical contact time bounded below by the limit [3]. To reduce an inertio-capillary time scale in symmetric bouncing, previous studies confirmed that the contact time can be altered below the time scale by physical structures, including macro-ridge and pillars. The impact dynamics of compound droplets on obstacle surfaces can offer potentials for further adjusting the dynamics by enhancing the asymmetry in the mass or momentum. The current study investigates the fundamentals of how compound droplets with different-viscosity components affect spreading, retraction, and bounce behavior on obstacle surfaces. The dynamics of the droplets were analyzed for different viscosity ratios, impact velocities, and shapes and sizes of the obstacles. We discuss the underlying mechanism behind the variation in the contact time by exploring the evolutions of the shape and axial momenta of the droplets. This study believes that the extraordinary bouncing of the droplets on surfaces decorated with obstacle can provide an insight into the effective regulation of the post-impact dynamics of viscous or rheological fluids in various applications.

Keywords: superhydrophobic surface, self-cleaning, interfacial dynamics

Acknowledgments:

This was supported by the National Research Foundation of Korea (NRF) grant funded by the Korea government (MSIT) (No. 2022R1C1C1003711).

References:

1. Richard, D., Clanet, C., Quéré, D. (2002). Contact time of a bouncing drop. *Nature*, 417(6891), 811-811.
2. Bird, J. C., Dhiman, R., Kwon, H. M., Varanasi, K. K. (2013). Reducing the contact time of a bouncing drop. *Nature*, 503(7476), 385-388.
3. Yun, S., Lim, G. (2014). Control of a bouncing magnitude on a heated substrate via ellipsoidal drop shape. *Applied Physics Letters*, 105(24).

On the use of a plasma jet for atomic oxygen production: Source characterization and application in cultural heritage

M. Poupouzas [1*], A. Nikiforov [2], R. Morent,[2] A. Sobota [1]
[1] Department of Applied Physics, EPG, Eindhoven University of Technology, 5600 MB Eindhoven, The Netherlands
[2] Research Unit Plasma Technology (RUPT), Department of Applied Physics, Ghent University, Sint-Pietersnieuwstraat 41, 9000 Ghent, Belgium

Abstract:

Non-thermal radio frequency (RF) driven Atmospheric Pressure Plasma Jets (APPJs) are efficient sources of reactive oxygen and nitrogen species (RONS)[1]. APPJs can be operated in ambient air, so that generated RONS can be effectively delivered onto target surfaces without need of complex vacuum system. This is e.g., useful for biomedical applications[2], liquid treatment[3] and deposition[4]. Recently our team started investigating the applications of plasma technology in cultural heritage[5] for the cleaning of different samples/artifacts. In a series of articles *Bruce Banks et al.* highlighted the importance of atomic oxygen in cleaning different samples (e.g. limestone, ivory, etc.) from soot contamination [6], [7].

The main source of atomic oxygen in He RF APPJ as described by *Waskoenig et al.* [8] is electron impact dissociation:

$$e^- + O_2 \rightarrow O + O(D) + e^- \quad (1)$$

and the following helium induced destruction process of oxygen metastables, $O(D)$:

$$He + O(D) \rightarrow O + He \qquad (2)$$

Upon meeting the surface, atomic oxygen reacts instantly with carbon/organic soiling, producing volatile by-products such as CO, CO_2 , and H_2O vapors.

Accordingly, in this work an RF atmospheric pressure plasma jet is used to induce atomic oxygen chemistry in order to treat various targets. The beam is produced by a coaxial electrode configuration. An RF discharge is established between a single axial pin electrode and the grounded stainless steel nozzle. The plasma operates in a mixture of He and O_2. By applying the gas flow the reactive species generated inside the nozzle are transported in the ambient air forming an effluent rich of atomic oxygen. As shielding gas, different noble gases such as Ar and He are used. The purpose of the shielding gas is the enhancement of O transport downstream to the sample and protection of the plasma chemistry from entrance and mixing with ambient air.

Within the context of this study, a parametric investigation of emission spectra is conducted by varying the ratio of the flows of main gas and shielding gas. Emission bands and lines such as OH at 309 nm, N_2 at 316, 337, 357, and 379 nm, O at 777.4, 844, 926 nm and He at 706.5 nm are observed in all experimental cases. The dynamics of O formation is studied with the use of OES. A special optical arrangement is applied to observe the plasma effluent radiation both radially and axially with a spatial resolution of 0.3 mm. The plasma input power is measured with a commercial RF power meter. A detailed schematic of our experimental set up is shown in Figure 1.

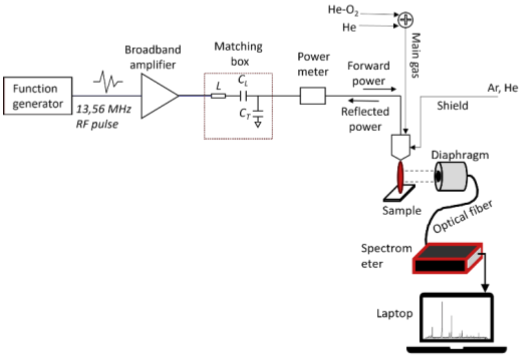


Figure 1: Conceptual diagram of the experimental setup.

The target consists of a glass slide fixed on top of a lab jack of micrometric precision placed at 2 mm from the nozzle tip. Plasma emission is probed both with and without the target to get insight on the effect of the target on excitation mechanisms.

In the range of parameters tested, we report a strong cleaning effect in several treated samples (see Figure 2).

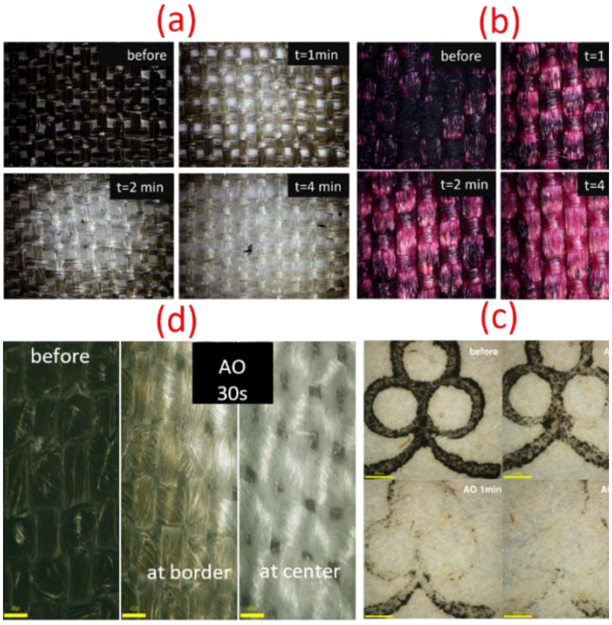


Figure 2: Samples: (a) Sooted silk blank, (b) Sooted silk cochineal, (c) Ink from paper, (d) Sooted linen blank.

Considering recent progress made by our team, a novel plasma source has yielded sufficient evidence of atomic oxygen plasma beam cleaning of CH relevant materials. With several limiting factors being the working power, treatment distance, etc., an in-depth study will be conducted to fully understand the plasma chemistry and cleaning process.

Keywords: APPJs, plasma-target interactions, Atomic Oxygen, RONS, Optical Emission Spectroscopy, Cultural Heritage.

Acknowledgements: This work is supported by the European Commission's Horizon Europe in the call Green technologies for Cultural Heritage: HORIZON-CL2-2021-HERITAGE-01-01 (grant agreement ID: 101061336).

References:

1. A. Schmidt-Bleker, J. Winter, S. Iseni, M. Dünnbier, K. D. Weltmann, and S. Reuter, "Reactive species output of a plasma jet with a shielding gas device - Combination of FTIR absorption spectroscopy and gas phase modelling," *J Phys D Appl Phys*, vol. 47, no. 14, Apr. 2014, doi: 10.1088/0022-3727/47/14/145201.
2. M. K. J. Nicol *et al.*, "Antibacterial effects of low-temperature plasma generated by atmospheric-pressure plasma jet are mediated by reactive oxygen species," *Sci Rep*, vol. 10, no. 1, Dec. 2020, doi: 10.1038/s41598-020-59652-6.
3. P. Svarnas *et al.*, "Water Modification by Cold Plasma Jet with Respect to Physical and Chemical Properties," *Applied Sciences (Switzerland)*, vol. 12, no. 23, Dec. 2022, doi: 10.3390/app122311950.
4. P. Luan, A. J. Knoll, H. Wang, V. S. S. K. Kondeti, P. J. Bruggeman, and G. S. Oehrlein, "Model polymer etching and surface modification by a time modulated RF plasma jet: Role of atomic oxygen and water vapor," *J Phys D Appl Phys*, vol. 50, no. 3, Jan. 2017, doi: 10.1088/1361-6463/aa4e97.
5. A. Patelli *et al.*, "A customised atmospheric pressure plasma jet for conservation requirements," in *IOP Conference Series: Materials Science and Engineering*, Institute of Physics Publishing, Jun. 2018. doi: 10.1088/1757-899X/364/1/012079.
6. S. K. Rutledge, B. A. Banks, V. A. Chichernea, and C. A. Haytas, "Cleaning of Fire Damaged Watercolor and Textiles Using Atomic Oxygen," 2000. [Online]. Available: http://www.sti.nasa.gov
7. B. A. Banks, S. K. Rutledge, and M. J. Norris, "An Atmospheric Atomic Oxygen Source for Cleaning Smoke Damaged Art Objects," 1998. [Online]. Available: http://www.sti.nasa.gov
8. I. Korolov *et al.*, "Atomic oxygen generation in atmospheric pressure RF plasma jets driven by tailored voltage waveforms in mixtures of He and O2," *J Phys D Appl Phys*, vol. 54, no. 12, Mar. 2021, doi: 10.1088/1361-6463/abd20e.

Slot-die coating of cellulose nanocrystals for improved barrier properties of paper

V. Kokol
[1] University of Maribor, Faculty of Mechanical Engineering, Slovenia; vanja.kokol@um.si

Abstract:

Surface coating with biopolymers is an emerging and the most common approach to improve barrier properties of paper to be used in various packaging applications (pharmaceuticals, food, medical), also following the circular economy and overall sustainability.

The combination of cellulose nanocrystals (CNCs) and cellulose nano- and microfibrils (CNFs, CMFs) without, or in combination with polysaccharides, such as chitosan, starch, pectin and alginate, have been studied extensively as candidates for high air and oxygen barrier coatings due to their high interaction with oxygen molecules, as well as matrices for the integration of other additives (such as antimicrobials, antioxidants, nutrients, etc) to enhance the papers` functionalities further.

Various coating techniques are employed to apply low-viscous biopolymers, including nanocellulose, either one or multiple coating layers onto the paper by using curtain coating, size press, rod/blade, and dip coatings. However, multiple layers (between 5 - 10) of deposition are required to cover the base paper's surface fully (generally between 10-15 g/m^2), thus to achieve desirable barrier effects. An uniform and relatively-higher coat weight (>10 g/m^2) at high coating speeds (up to several 10 m/min) can be achieved only by curtain or slot-die coating process, as well as blade coating, leading to complete coverage of the paper's surface. On the other hand, the bar/rod coating technique provides better control over the coating thicknesses, however, it is confined only to the laboratory or pilot scale, and not considered for scale-up. In the case of the dip coating process, it is challenging to control the coating thickness.

Both low and high viscosity solutions, including CNF and MFC suspensions, have been tested in a slot-die coating technique.

In this work, the slot-die deposition technique was thus used to apply a moderately concentrated CNCs` suspension onto the paper by a fully automated (computer controlled) laboratory coating device employing high performance near-infrared (NIR) energy drying. In contrast to the conventional drying technologies that have to heat up the substrate surface before the energy also reaches the water molecules in it, the electromagnetic waves of **NIR** radiation can penetrate quickly (in a few sec instead of min by using hot air) and deeply into the coated substrate in the smallest applicable areas. Such an evenly performed heating can activate water molecules directly, and removes them from the entire thickness with extremely short processing times. NIR heating can thus reduce the energy consumption, especially in continuous manufacturing processes like paper industries, that require continuous drying. However, no study is yet available on how effective such a highly intensive heating method would be in the drying of biopolymer coated suspensions, nor how it would affect their integrity, uniformity and adhesion to the paper`s surface.

The effect of the established coating parameters (injection speed and dry thickness settings, at a given slot gap thickness and slot-to-paper distance) was investigated by the coating quantity and homogeneity, including the papers` physical (grammage, thickness, density) properties and whiteness. The effect of the papers` structure on the coating adhesion was determined, as well as their barrier (air, oxygen, water vapour permeability) properties.

Keywords: Paper; Nanocellulose; Slot-die coating; NIR drying; Barrier properties.

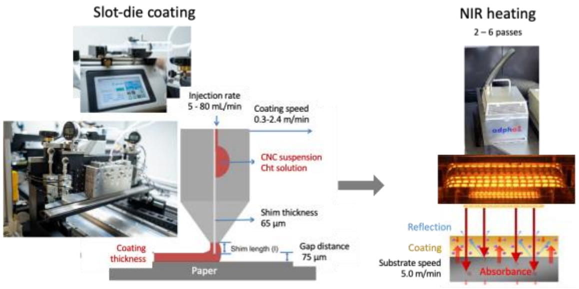


Figure 1: Schematic presentation of the equipment with the coating experiment`s set-up that was used.

Reference:

Ruberto Y, Vivod V, Juhant Grkman J, Lavrič G, Graiff C, Kokol V, Slot-die coating of cellulose nanocrystals and chitosan for improved barrier properties of paper, submitted to *Cellulose*.

Atmospheric pressure glow discharge for the determination of hydride-forming elements

K. Gręda [1,*], J. Kratzer [2], M. Svoboda [2], N. Vlčková [2], J. Dědina [2], P. Pohl [1]
[1] Division of Analytical Chemistry and Chemical Metallurgy, Wroclaw University of Science and Technology, Wroclaw, Poland
[2] Department of Trace Element Analysis, Institute of Analytical Chemistry of the Czech Academy of Sciences, Prague, Czech Republic

Abstract:
Chemical hydride generation (HG) was combined with a direct current – atmospheric pressure glow discharge (dc-APGD) to sensitively quantify As, Sb, and Se by optical emission spectrometry (OES). The discharge was operated in a gas jet, the total power consumption was 15 W, and the plasma volume was approximately 8 mm^3 (power density about 2W/mm^3). It was revealed that plasma operated in He was much more stable than Ar-plasma. The carrier gas with the analyte hydrides produced by HG and together with co-generated concomitant H_2 was introduced into the dc-APGD plasma, where atomization and excitation processes took place. It was found that the presence of H_2 in the discharge gas significantly reduced the intensity of NO and N_2 molecular bands. Under the optimized operating conditions, the measurement repeatability was 2-3%, proving the high stability of the whole HG-dc-APGD OES system. The limits of detection (LODs) for As, Sb, and Se reached 0.6, 0.14, and 1.2 µg L^{-1}, respectively, and they were lower than those routinely reported for inductively coupled plasma (ICP) OES. In comparison to the commonly used ICP OES instruments, the novel microplasma system offered a simple and more compact design. The trueness of HG-dc-APGD OES was verified by a recovery test. High agreement between spiked and measured concentrations of As, Sb, and Se in mineral and tap water samples was obtained (recoveries within the range of 96-103%), which proves the high accuracy of the developed method.

Keywords: atmospheric pressure plasma, glow discharge, microplasma, optical emission spectrometry, detection of toxic elements.

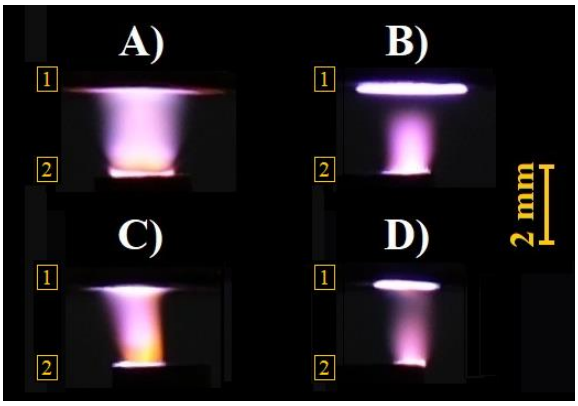


Figure 1: APGDs operated under different conditions. A) He-APGD, nozzle as cathode, B) He-APGD, nozzle as anode, C) Ar-APGD, nozzle as cathode, D) Ar-APGD, nozzle as anode.
[1] – Solid electrode. [2] – Gas nozzle.

References:

1. Gręda, K., Kratzer, J., Svoboda, M., Vlčková N., Dedina, J., Pohl, P. (2024) A novel atmospheric pressure glow discharge system for sensitive determination of As, Sb, and Se by optical emission spectrometry followed their hydride generation, *Spectrochim. Acta B,* Under Revision.

Acknowledgments: This research has been supported by a bilateral research project evaluated under a Lead Agency principle. Pawel Pohl and Krzysztof Greda acknowledge financial support from the Polish National Science Center (2022/04/Y/ST4/00055) while the other authors are grateful to the Czech Science Foundation (23-05974K) and the Czech Academy of Sciences, Institute of Analytical Chemistry (Institutional Research Plan no. RVO: 68081715) for financial support.

Instabilities in O_2 Capacitively Coupled Radio-Frequency Plasmas

M. Myrzaly[1,2*], R. Masheyeva[2,3], C. Tian[4,5], M.Vass[3,5], L.Y. Luo[6], K. Dzhumagulova[1,2], P. Hartmann[3], J. Schulze[5], Z. Donkó[3]
[1]Research Institute of Experimental and Theoretical Physics, Al-Farabi Kazakh National University, Almaty, Kazakhstan
[2]Department of General Physics, Satbayev University, Almaty, Kazakhstan
[3]HUN-REN Wigner Research Centre for Physics, Budapest, Hungary
[4]Key Laboratory of Materials Modification by Laser, Ion, and Electron Beams (Ministry of Education), School of Physics, Dalian University of Technology, Dalian, China
[5]Chair of Applied Electrodynamics and Plasma Technology, Department of Electrical Engineering and Information Sciences, Ruhr University Bochum, Bochum, Germany
[6]Department of Engineering Physics, Tsinghua University, Beijing, China

Abstract:

Low-temperature capacitively coupled plasmas (CCPs) find widespread applications in diverse industries, ranging from microelectronics to biomedical fields. In particular, industrial processes often utilize electronegative gases such as Cl_2, CF_4, SF_6, and O_2. The presence of negative ions in the discharge significantly alters the plasma physics, leading to the development of various instabilities related to the electronegativity of the gas [1-4]. Understanding the development of these instabilities is crucial for optimizing and controlling low-temperature CCPs in various industrial applications.

This study focuses on the investigations of kHz instabilities in oxygen (O_2) plasmas. The experiments, conducted in a Budapest Cell v.3 discharge system [5], serve as crucial reference data for ongoing Particle-in-Cell/Monte Carlo Collision (PIC/MCC) simulations.

The experiments cover gas pressures ranging from p = 50 Pa to 400 Pa. From the measurement of the light emission distributions at a fixed pressure and different voltages in the range of V_{pp} = 500 V to 1400 V, it has been observed that the light emission intensity increases monotonically with the discharge voltage (and power) at each position. Additionally, for voltages $V_{pp} > 800$ V, an additional maximum appears in the intensity profile close to the electrode surfaces, at a position that is independent of the voltage and pressure [4].

Periodic oscillations are observed by gradually increasing the voltage for each fixed value of the pressure. These oscillations disappear at a certain value of the voltage. It has been observed that there is no instability below p = 50 Pa and that the oscillation frequency is found to be pressure-dependent. Additionally, the pulse characteristics change as the voltage increases within the instability region. The results can provide valuable insights into the dynamics of kHz instabilities in oxygen discharges, serving as a benchmark for numerical simulations.

This work was supported by the grant AP19679536 from the Ministry of Higher Education and Science of the Republic of Kazakhstan.

Keywords: capacitively coupled plasma, electronegative gases, attachment-induced ionization instability.

References:

1. Nighan, W.L. and Wiegand, W.J. (1974) Influence of negative-ion processes on steady-state properties and striations in molecular gas discharges, *Phys. Rev. A*, 10, 922–945.
2. Descoeudres, A., Sansonnens, L, and Hollenstein, Ch. (2003) Attachment-induced ionization instability in electronegative capacitive rf discharges, *Plasma Sources Sci. Technol.*, 12(2), 152.
3. Küllig, C., Wegner, Th. and Meichsner, J. (2015) Instabilities in a capacitively coupled oxygen plasma, *Phys. of Plasmas*, 22(4), 043515.
4. Dittmann, K., Matyash, K., Nemschokmichal, S, Meichsner, J., and Schneider R. (2010) Excitation mechanisms and sheath dynamics in capacitively coupled radio-frequency oxygen plasmas, *Contr. Plasma Phys.*, 50(10), 942–953.
5. Horváth, B., Derzsi, A., Schulze, J., Korolov, I., Hartmann P. and Donkó Z. (2020) Experimental and kinetic simulation study of electron power absorption mode transitions in capacitive radiofrequency discharges in neon. *Plasma Sources Sci. Technol.,* 29 055002 .

Study of the surface and effect of cold plasma treatment on the adhesion of Polyphenylene Sulfide

M.A. Martinez[1*], J. Abenojar[1,2]
[1] Department Material Science and Engineering. University Carlos III of Madrid, Spain
[2] Department Mecanical Engineering. University Pontificia Comillas of Madrid, Spain

Abstract:

Polyphenylene sulfide (PPS) stands as a prevalent engineering plastic, renowned for its high-performance attributes as a semi-crystalline thermoplastic. Comprising aromatic rings bonded by sulfides, PPS exhibits a brittle structure, boasting a high glass transition temperature and melting point. Its mechanical strength aligns seamlessly with outstanding thermal and chemical resistance, coupled with commendable electrical properties. Remarkably, PPS is non-combustible and self-extinguishing, making it an ideal polymer for crafting components enduring elevated temperatures and chemical exposure—think pump parts, fans, and impellers in the automotive sector, encompassing pumps, valves, air supply system elements, seals, carburetors, and heating, ventilation, and air conditioning system components.

This study undertakes the characterization of PPS surface properties and explores its adhesion with various adhesives, notably cyanoacrylate and silicone. Additionally, the investigation delves into the impact of atmospheric pressure plasma surface treatment (APPT) on the material's surface and potential adhesion enhancement. Optimizing APPT parameters involves maintaining a 10 mm distance from the sample surface to the torch and a torch rate of 10 mm/s. Post-treatment, the free surface energy (SE) experiences a 37% increase, with the polar SE component soaring by 93% (Figure 1). However, the treatment's durability diminishes after 48 hours, prompting adhesive bonding with cyanoacrylate (HY4070) and silicone (WS-605S) within the initial 24 hours of APPT.

APPT improves adhesion with cyanoacrylate by 77% and with silicone by 94%, as evidenced in pull-off tests. Concurrently, the study explores shot-blasting as an alternative treatment for PPS, revealing an increase in SE attributed to the dispersive component (Figure 1), what does not improve the adhesion with these adhesives.

Keywords: Polyphenylene sulfide, Plasma treatment, shot-blanding treatment, Surface energy, adhesion, cyanoacrylate, silicone.

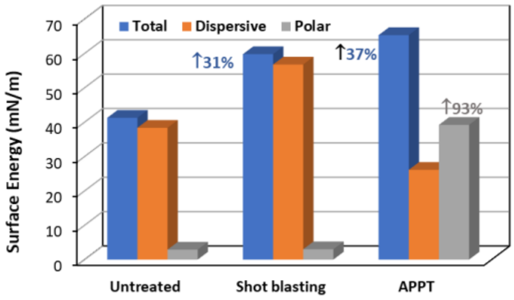


Figure 1: PPS surface energy before treatment and after shor blasting and APPT treatments.

References:

1. Zuo, PY., Tcharkhtchi, A., Shirinbayan, M., Fitoussi, J., Bakir, F. (2019) Overall investigation of polyPhenylene sulfide (PPS) from synthesis and process to applications-A review. *Macromol Mater Eng* 304(5), 1800686.
2. Martinez, M.A., Abenojar, J., Lopez de Armentia, S. (2018) Environmentally friendly plasma activation of acrylonitrile-butadiene-styrene and polydimethylsiloxane surfaces to improve paint adhesion. *Coatings* 8/10, 428.

Hierarchically Nanostructured metal oxide thin films by plasma

C. Riccardi, H.E. Roman
Dipartimento di Fisica G. Occhialini, Università degli Studi di Milano-Bicocca,
piazza della Scienza 3, I-20126 Milano, Italy

Abstract

The controlled growth of nanostructured thin films represents a challenging field of research which is related to many different applications of great scientific relevance. The properties of many materials can be greatly enhanced by optimising the nanoscale assembly processes: it requires a bottom-up approach capable of tailoring the properties with a high level of control or a complex set-up. Plasma-based synthesis processes have been widely developed and applied for an increasing number of technologies leading to important achievements and many industrial-scale applications, in particular in the field of nanoscience.

Combining a reactive plasma with a supersonic jet we developed thin film deposition hierarchically nanostructured can be performed: [1]. An argon-oxygen inductively coupled plasma offers a reactive environment where metalorganic precursors are dissociated and oxidised. The gas is then left to expand from a small orifice into a lower pressure vacuum vessel forming a supersonic jet, where the nanoparticles are assembled and accelerated onto a substrate by the gas carrier mixture. This deposition technique has proved useful for the deposition of nanostructured thin film having a desired morphology at competitive deposition rates.

References

1. Dell’Orto, E. C.; Caldirola, S.; Sassella, A.; Morandi, V.; Riccardi, C. Growth and Properties of Nanostructured Titanium Dioxide Deposited by Supersonic Plasma Jet Deposition. *Appl. Surf. Sci.* 2017, *425*, 407–415.
2. Hierarchically Carbon Nanocone-Silica Metamaterials: Implications for White Light Photoluminescence, Carra, C. et al., ACS Applied Nano Materials, 2022, 5(4), pp. 4787–4800
3. Controlled Deposition of Nanostructured Hierarchical TiO2 Thin Films by Low Pressure Supersonic Plasma Jets, Piferi, C., Nanomaterials, 2022, 12(3), 533

Fabrication of Atmospheric Plasma-reduced rGO-based Nanocomposites and Its Applications

František Zelenák [1,*], Richard Krumpolec [1], Jana Jurmanová [1], Monika Stupavská [1],
Mária Kováčová [2], Pavel Neilinger [3], Zdeněk Moravec [4], Miroslav Grajcar [3], Mirko Černák [1]
[1] Department of Plasma Physics and Technology, CEPLANT—R&D Center for Plasma and Nanotechnology Surface Modifications, Faculty of Science, Masaryk University, Kotlářská 2, 611 37 Brno, Czech Republic
[2] Department of Composite Materials, Polymer Institute, Slovak Academy of Sciences, Dúbravská cesta 9, 845 41, Bratislava 45, Slovak Republic
[3] Department of Experimental Physics, Faculty of Mathematics, Physics and Informatics, Comenius University in Bratislava, Mlynská dolina, 842 48 Bratislava, Slovak Republic
[4] Department of Chemistry, Faculty of Science, Masaryk University, Kamenice 5, 625 00 Brno, Czech Republic

Abstract:

A top-down approach by oxidation of graphite to graphene oxide (GO) and its reduction via various techniques gained popularity for a mass production of thin conductive graphene-like films. The most advantageous alternative to pure graphene is reduced graphene oxide (rGO) as it is almost alike graphene in terms of properties and can be fabricated from GO using several reduction/exfoliation techniques. The known reduction/exfoliation techniques, however, have limitations such as high levels of toxicity, hazardousness, risk of explosions, and high time and energy consumption. The complicated reduction/exfoliation of GO become a major bottleneck that restricts the large-scale production of rGO. The research team of CEPLANT have developed a simple, fast, low-cost and chemical-free fabrication method of 2D and 3D rGO structures using an electrical plasma-triggered reduction&exfoliation of 3D porous GO aerogel-like materials [1] and also 2D GO paper-like films [2] starting at temperatures not higher than 100 °C.

The direct exposure of self fabricated GO structures to low-temperature atmospheric plasma generated by the Diffuse Coplanar Surface Dielectric Barrier Discharge (DCSBD) in technical nitrogen or standard air, triggers the self-propagating reduction&exfoliation modification of GO into the rGO. The rapid modification of GO into rGO do not result in abrupt disintegration of the sample and led in an approximately 10^5-fold decrease of the rGO resistivity compared to the original GO.

This novel plasma-triggered reduction&exfoliation method opens new opportunities for low-cost industrial-scale production of conductive rGO and rGO-based composites for various applications. We will introduce the challenges of impregnation methods to improve the mechanical properties of highly-porous 3D rGO structures as well as 2D rGO papers (Figure 1).

Finally, we will show how fabricated rGO-based composites can be used for particular applications in the field of i) electromagnetic shielding; ii) sensors, iii) energy storage, and iv) water and air treatment.

Keywords: atmospheric plasma, DCSBD, low-temperature, graphene oxide, reduced graphene oxide, self-propagating reduction, exfoliation, impregnation, composites, applications.

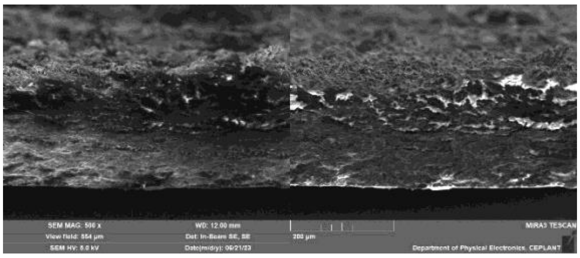


Figure 1: Figure of cross-sectional SEM view of drop-coating impregnated 2D rGO paper.

References:

1. Krumpolec, R., et al. (2022), High conductive rGO sheets fabricated by mild, low-cost and scalable plasma-triggered reduction-exfoliation of 3D aerogel-like graphene oxide, *FlatChem,* 35, 100403.
2. Zelenák, F., et al. (2023), Fast, scalable, and environmentally friendly method for production of stand-alone ultrathin reduced graphene oxide paper, *Carbon,* 215, 118436.

Effect of molybdenum concentration and deposition temperature on the structure and tribological properties of the diamond like-carbon films.

H. Zhairabany*[1], A. Sarakovskis[2], E. Gnecco[3], E. Vanags[2], L. Marcinauskas[1]

[1] Department of Physics, Kaunas University of Technology, Studentų str. 50, Kaunas, Lithuania
[2] Institute of Solid State Physics, University of Latvia, Kengaraga Str 8. Riga, Latvia.
[3] M. Smoluchowski Institute of Physics, Jagiellonian University, ul. Lojasiewicza 11, Krakow, Poland.

Abstract:
Nowadays, in the diamond-like carbon (DLC) films synthesis several metals such as Mo, Ti, Cr, Ag, etc. are used as dopants [1], in order to modify the film structure and as consequence enhancing a desirable properties of DLC thin films. It was demonstrated that the Mo-doped DLC films have demonstrated better tribological properties and enhanced mechanical characteristics. It should be noted that the final properties of the Mo-DLC films strongly depend on the Mo concentration, synthesis conditions or type of used gas [2-4]. The objective of this work was to deposit and characterize the microstructure and tribological properties of DLC films with various Mo content and determine the influence of the deposition temperature on the properties of the Mo-DLC films.

Molybdenum-doped amorphous diamond-like carbon thin films (Mo-DLC) were deposited on Si (100) substrates by direct current magnetron sputtering. The graphite and the molybdenum cathodes were used. The arc currents were fixed at 1.0 A and 0.25 A for graphite and molybdenum targets, respectively. The temperature of formation was changed between 170-240 °C by changing the target-substrate distance between 4-8 cm. The Mo content was changed by increasing the opening of a shield mounted above the molybdenum target. The elemental composition, surface morphology, structure, nanohardness and friction forces of the Mo-DLC films were investigated by energy dispersive X-ray spectroscopy (EDS), Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM) and nanoindenter.

The results indicated that the Mo content increased with the opening of the shield above the Mo target. The Mo concentration in the films was from 1 at. % up to 6.6 at.%. It was obtained that the increase of the Mo amount increased the oxygen concentration in the DLC films. Additionally, the Raman spectra of the films indicated that the sp^2 carbon sites fraction increased, and the graphitization was induced with the increase of the deposition temperature. The XPS results revealed a small amount of Mo carbide phase formation in the films. The fraction of the MoC sites slightly increased with the increase of Mo content. The friction coefficient of the Mo-doped DLC films depended on the deposition temperature and composition of the films. It was found that the Young's modulus and the nanohardness of the Mo-DLC films was enhanced with the addition of low amount of Mo. The nanohardness of the films was enhanced with the reduction of the deposition temperature.

Keywords: Molybdenum, diamond-like carbon, microstructure, tribology, friction coefficient, nanohardness.

References:

1. Khrushchov M, Levin I, Marchenko E, Avdyukhina V, Petrzhik M (2016) Effect of structure and deposition technology on tribological properties of DLC coatings alloyed with VIA group metals. J Phys Conf Ser 729:012016.
2. Padmanaban DB, Mohan L, Giri P, Bera P, Anandan C, Barshilia HC (2020) Effect of molybdenum content on mechanical and tribological properties of diamond-like carbon coatings over titanium β-21S alloy. C (Basel) 7(1):1.
3. Yongyao S, Xiaohong G, Weijiu H, Tengfei Z, Rong H, Peng Z, Haibo R, Yucheng M (2023) Enhancing the tribological property of Mo-doped DLC films in methanol using appropriate bias voltage. Diamond and Related Materials, Volume 135, ISSN 0925-9635.
4. Yongyao S, Weijiu H, Lixin C, Xiaohong G, Tengfei Z, Rong H, Peng Z, Haibo R (2022) Microstructural evolution and tribology of Mo-doped diamond like carbon nanocomposite film. Tribology International, Volume 174, ISSN 0301-679X.

Towards green tribology: water versus oil lubrication of laser-textured Ti6Al4V alloy upon addition of MoS2 nanotubes

M. Conradi[1*], B. Podgornik[1], A. Kocijan[1], M. Remškar[2], D. Klobčar[3]
[1]Institute of metals and technology, Lepi pot 11, 1000 Ljubljana, Slovenia
[2]Jozef Stefan Institute, Jamova 39, 1000 Ljubljana
[3]Faculty of Mechanical Engineering, University of Ljubljana, Aškerčeva 6, 1000 Ljubljana, Slovenia

Abstract:

A Nd-YAG laser was used for texturing the Ti6Al4V surface with dimples of diameter 50 and 100 µm and centre-to-centre distance 100, 200 and 400 µm, defining the surface texture density. Tribological evaluation was conducted to analyse and compare the behaviour of un-textured and laser-textured samples under water in comparison to oil (PAO6) lubrication without and with the addition of MoS_2 nanotubes into the lubricant. MoS_2 nanotubes had positive effect on friction in both media for laser-textured Ti6Al4V. The best results regarding friction and wear were found on the 100-200 (diameter-center-to-center distance) surface lubricated by PAO6 oil with 2 wt. % MoS_2, where low wear rates of untextured surface were maintained while friction was reduced for more than 30 %. For water, the best tribological results were obtained for the 100-400 pattern in terms of wear and 50-100 in terms of fric-tion upon addition of 2 wt. % MoS_2.

The current research confirms that water lubrication is competitive to oil lubrication for specific laser-textured configurations of Ti6Al4V surface, especially upon addition of MoS_2 nanotubes as solid lubricant. This suggests that laser-textured Ti6Al4V alloy can be used also in tribologically challenging environments using water as lubricant. The application of water-based lubrication with MoS2 nanotubes is therefore within the concept of green tribology, which focuses on balancing between the tribological and ecological as-pects with minimal environmental and biological impact. This offers the possibility to implement Ti6Al4V alloy in more tribologically challenging working environments.

Keywords: green tribology; surface texturing; MoS_2 nanotubes; lubrication; Ti6Al4V alloy.

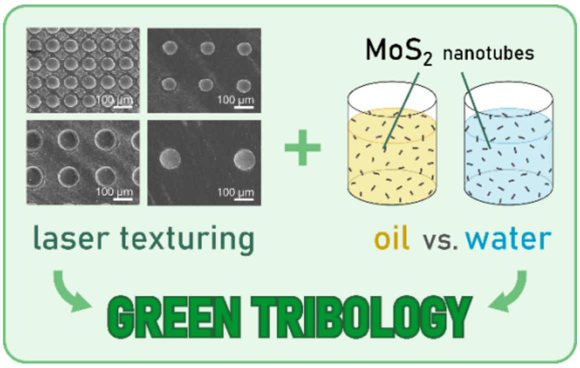


Figure 1: Laser-textured surfaces towards green tribology: Oil vs. water lubrication with the addition of MoS_2 nanotubes.

References:

1. Conradi, M., Podgornik, B., Kocijan, A., Remškar, M., Klobčar, D. (2022) Water versus oil lubrication of laser-textured Ti6Al4V alloy upon addition of MoS_2 nanotubes for green tribology, *Materials*, 15, 1-12.
2. Nosonovsky M, Bhushan B. (2010), Green tribology: principles, research areas and challenges, *Trans R Soc A*, 368, 4677–4694.

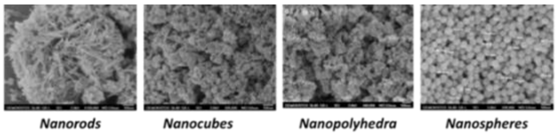
The effect of shape and size of nanoceria on the antimicrobial properties of nanocoatings

Z. Sideratou[1], A. Papavasiliou[1], K.M. Lyra[1], D. Tsiourvas[1], L. Hernández Ruiz[2], T. Oroz Mateo[2], F.K. Katsaros[1]
[1]Institute of Nanoscience and Nanotechnology, NCSR “Demokritos”, Aghia Paraskevi Attikis, Athens 15310, Greece
[2]Centro Tecnológico Lurederra, Industrial Area Perguita, C/A N° 1 31210, Los Arcos, Navarra, Spain

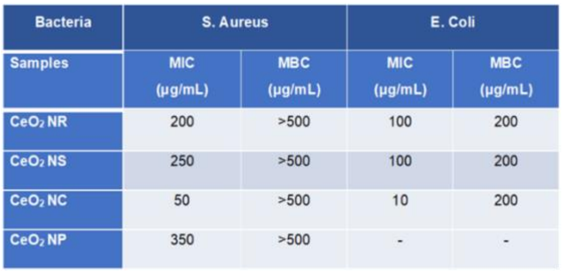
Abstract:

Infectious diseases pose a significant threat to human health and the global economy, accounting for more than 20% of global mortality, while viruses are responsible for about one-third of these deaths. To date, about 200 infectious diseases and about 80% of infectious diseases are transmitted by hands touching contaminated surfaces. To tackle with these threats, antimicrobial coatings are widely used. Although their benefits, there are certain drawbacks of the current State-of-the-art level related to: (i) the release of the active compounds that might slowly enter the ecosystem, (ii) the loss of their efficiency in short time, (iii) the resistance of the microbes to the commonly used agents and (iv) the high human toxicity of some antimicrobial agents (e.g., Ag). Addressing these challenges, the current work is focussed on the development of new sustainable antiviral and antimicrobial coatings for textiles and high traffic objects made of plastics and metal, involving bathroom and switches manufacturing industries. In this context, the efforts were focused on the synthesis of novel active nanomaterials (ANMs) based on cerium oxide. Thus, ceria NPs with different sizes, morphologies and exposed crystal facets were developed. Nanoceria can act as an excellent antioxidant agent and protect the healthy cells from oxidative stress by scavenging reactive oxygen species (ROS) or as a pro-oxidant and hence exhibit a strong antibacterial effect[1]. The developed ANMs were characterised by a variety of technuques inclusing SEM, nitrogen porosimetry, XRD etc, while their antibacterial performance was evaluated according to the standards using *E.Coli* and *S. Aureus*. Rearding the effect of shape, the antimicrobial efficiency followed the order: nanocubes> nanorods> nanospheres> nanopolyhedra. The developed ANMs were incorporated in various matrices and used for antimicrobial coatings. Prior the application, Atmospheric plasma jet treatment was used to improve the surface adhesion of the coatings on the selected specimens.

Keywords: Active nanomaterials, nanoceria, gram negative and positive bacteria, coatings, atmospheric plasma treatment, antimicrobial efficiency.



Nanorods *Nanocubes* *Nanopolyhedra* *Nanospheres*



Bacteria	S. Aureus		E. Coli	
Samples	MIC (μg/mL)	MBC (μg/mL)	MIC (μg/mL)	MBC (μg/mL)
CeO_2 NR	200	>500	100	200
CeO_2 NS	250	>500	100	200
CeO_2 NC	50	>500	10	200
CeO_2 NP	350	>500	-	-

Figure 1: SEM images of ceria NPs with different shapes and antimicrobial efficiency on S. Aureus and E. Coli.

References:

1. Wong, C.S.-A., Lim, C.W., Mohammed H.I., Liew, K.Y., Tham, C.L., Tan, J.W., Chee, H.Y. (2022), Current Perspective of Plant-Based Diets on Communicable Diseases Caused by Viruses: A Mini Review, *Front. Nutr.* 9, 786972.
2. Thakur, N., Manna, P., Das, J. (2019), Synthesis and biomedical applications of nanoceria, a redox active nanoparticle, *J. Nanobiotechnol* 17, 84

Acknowledgements: This work funded by the European Union Horizon Europe Programme for Research and Innovation under the Grant Agreement N° 101057988 – SUSAAN.

Development of nanoporous nickel oxide materials as electrodes for supercapacitors

Yang-Ming Lu [1*], Yen-Ching Lin[2], Liu Ting Yi
Department of Electrical Engineering, National University of Tainan, Taiwan, R. O. C.

Abstract:
A supercapacitor is known for its properties of low weight, excellent power density, outstanding charging rate, and long durability. The choice of electrode material and its morphology significantly impact the overall performance of the supercapacitor. On the other hand, nickel oxide is a promising material due to its low cost, environmentally friendly nature, and high specific capacitance. In this research, we present a simple and cost-effective method for fabricating supercapacitor electrodes. We achieve this by using electrospinning and thermal annealing techniques, which allow for the direct growth of porous nickel oxide nanofibers on 3D-nickel foam. In this study, we have successfully fabricated binder-free electrodes using porous NiO nanofibers directly electrospun onto a Ni foam. These electrodes exhibited a high specific capacitance of 591 F/g at a current density of 2 A/g and demonstrated good cycling stability, with 87% retention after 1500 cycles. These results suggest that this electrode has the potential to be used as a candidate for supercapacitors.

Keywords: supercapacitor, nickel oxide , electrospinning , 3D-nickel foam

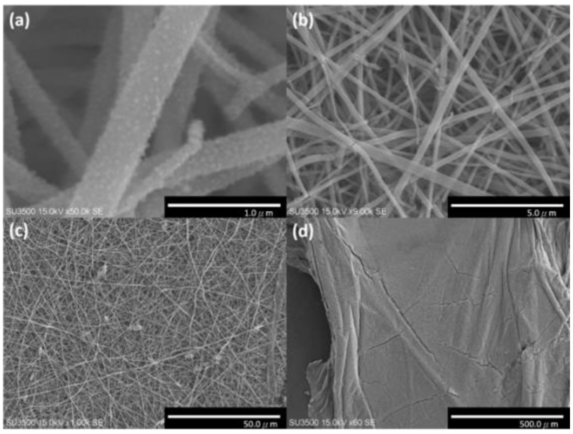


Fig. 2 SEM of the porous NiO-NFs electrospun on Ni foam at different magnifications (a) x50.0k (b) x9.0k (c) x1.0k (d) x60

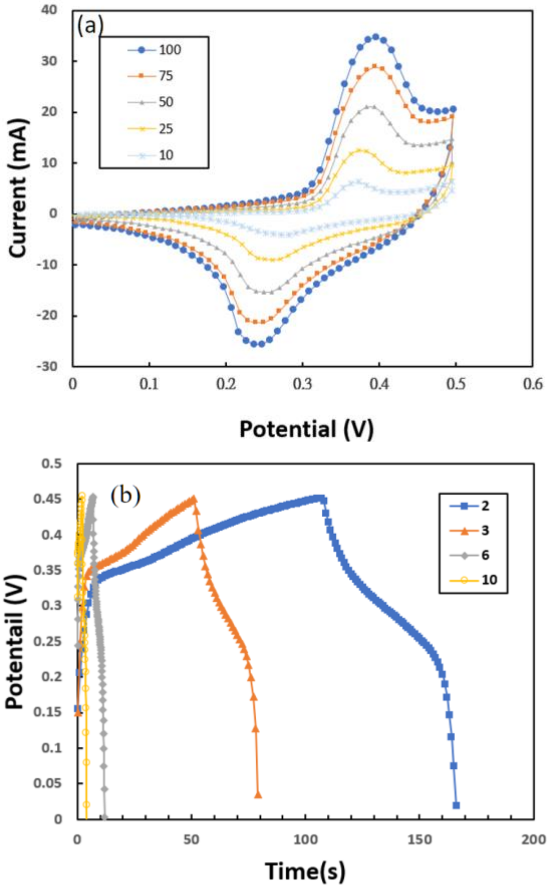


Fig. 2 The performances of (a) cyclic voltammetry (b) galvanostatic charge/discharge

References:

1. Lamastra, F. R., Nanni, F., Menchini, F., Nunziante, P., & Grilli, M. L. (2016). Transparent nanostructured electrodes: Electrospun NiO nanofibers/NiO films. Thin Solid Films,601,54–58. doi:10.1016/j.tsf.2015.09.019
2. Kundu, M., & Liu, L. (2015). Binder-free electrodes consisting of porous NiO nanofibers directly electrospun on nickel foam for high-rate supercapacitors. Materials Letters, 144, 114–118. doi:10.1016/j.matlet.2015.01.032

Saving Energy in Electrochemical Water Treatment Using BDD Electrodes Through Forced Fouling-Reactivation Cycles: Optimizing Frequency and Duty Cycle

P. Brosler [1], R. F. Silva[1], J. Tedim[1], F. J. Oliveira[1,*]
[1] CICECO - Aveiro Institute of Materials & Department of Materials and Ceramic Engineering, University of Aveiro, 3810-193 Aveiro, Portugal

Abstract:

This study introduces a groundbreaking approach to enhance the energy efficiency of electrochemical water treatment, focusing on the degradation of phenolic compounds. The method revolves around cyclic fouling and reactivation sequences applied to boron-doped diamond (BDD) electrodes, employing a two-step square wave pattern. Our investigation emerged from observations during chronoamperometry experiments with BDD electrodes in phenol-containing solutions, where cyclic variations in current density responses were noted, particularly at potentials above 4 V/SCE. The multi-step process of electrode fouling, induced by phenolic compounds, unfolds when a potential below the water stability region is applied. This induces the polymerization of phenoxy radicals, leading to the formation of a polymeric layer on the electrode surface. Our hypothesis builds on the cyclic variations observed, suggesting that the accumulation of phenolic compounds on the BDD electrode surface through electropolymerization at low applied potentials could be followed by applying a suitably elevated potential. This elevated potential is designed to detach the polymeric layer while simultaneously initiating its partial or complete oxidation. The study employs a comprehensive three-level full factorial experimental design to fine-tune the frequency and duty cycles of the square wave pattern, optimizing the fouling and reactivation steps. Results demonstrate that the proposed approach outperforms conventional continuous anodic oxidation processes, significantly reducing energy consumption by over four times while achieving superior phenol degradation. The novel square wave anodic oxidation technique required only 50 $\text{kWh/kg}_{\text{Phenol}}$ to achieve an 88% removal rate for a Na_2SO_4 solution containing 1mM phenol after one hour of treatment. In comparison, continuous anodic oxidation required 209 $\text{kWh/kg}_{\text{Phenol}}$ for an 83% removal rate under the same conditions. Investigating the broader applicability of the proposed fouling-reactivation strategy holds promise for addressing a broader range of persistent organic pollutants, thus contributing to the development of more sustainable water treatment solutions.

Keywords: water treatment, electrochemical oxidation, phenol, boron-doped diamond, fouling, energy efficiency, anodic oxidation

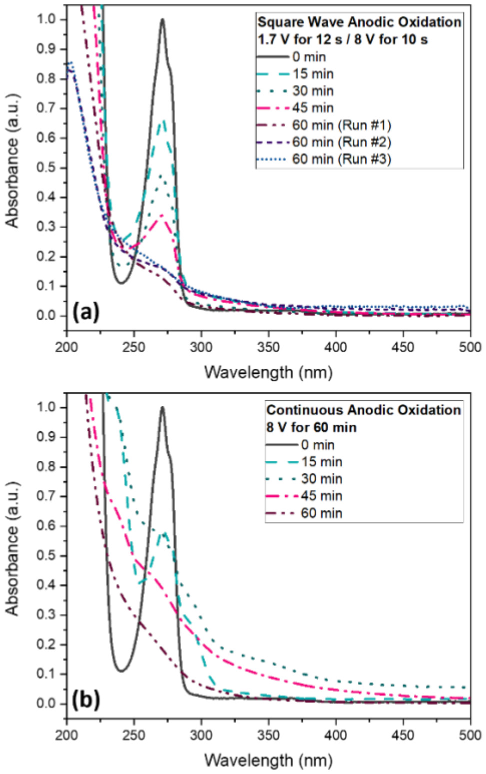


Figure 1: UV-Vis spectroscopy illustrating the phenol degradation progression over a 1-hour duration in two distinct assays: (a) Novel square wave anodic oxidation approach. (b) Conventional continuous anodic oxidation at 8V/SCE.

References:

1. Liu X., You S., Ma F., Zhou H. (2020), Characterization of electrode fouling during electrochemical oxidation of phenolic pollutant, *Front. Environ. Sci. Eng.*, 15, 4.
2. Zhi J.F., Wang H.B., Nakashima T., Rao T.N., Fujishima A. (2003), Electrochemical Incineration of Organic Pollutants on Boron-Doped Diamond Electrode. Evidence for Direct Electrochemical Oxidation Pathway. *J. Phys. Chem. B*, 107, 48.

Effect of plasma on cork particles to adsorb magnetite, and later, hexavalent chromium from water.

J. Abenojar[1,2,*], S. López de Armentia[3], M.A. Martínez[1], J.C. del Real
[1] Department Material Science and Engineering. University Carlos III of Madrid, Spain
[2] Department Mecanical Engineering. University Pontificia Comillas of Madrid, Spain
[3]IIT. Department Mecanical Engineering. University Pontificia Comillas of Madrid, Spain

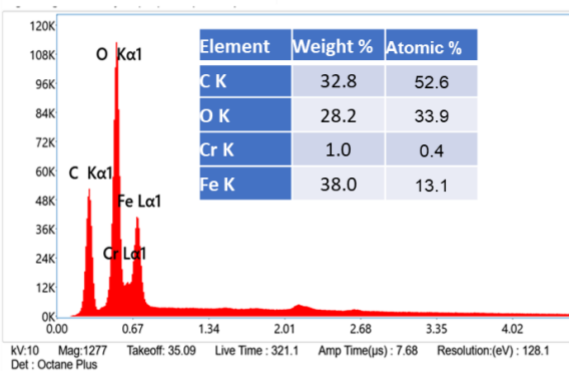
Abstract:

Cork, known for its hydrophobic properties with a surface energy (SE) of 18 mN m^{-1}, in spite of its porous state, maintains a low SE due to extractives, together with lignin, suberin, and polysaccharides. These extractives, whether polar or nonpolar, play a crucial role. Previous research indicates that plasma surface treatments significantly boost cork sheet SE by 2.5 to 3 times. In particular, low pressure plasma (LPP) enhances the polar SE component from 0 to 17 mN m^{-1}. This increase coincides with improved wettability, resulting from chemical cleaning and activation of the plasma. Cork's cellular structure, reminiscent of a honeycomb, undergoes changes post-LPP treatment. Cell walls become thinner and rounder, leading to reduced density and enlarged open honeycomb cells. This alteration increases the specific surface area, enhancing availability for adsorption.On the other hand, cork is a light weight, renewable and biodegradable material, gaining popularity in various industries, including its application as a Cr(VI) adsorbent in wastewater, competing with activated carbon and magnetite. On the other hand, cork is a light weight, renewable and biodegradable material, which is increasingly used in industry. One of its applications is as an adsorbent for Cr(VI) in wastewater. Other adsorbents used are activated carbon and magnetite.

This study's primary focus is to create magnetic cork for efficient Cr(VI) adsorption. The proposed approach involves an LPP treatment with specific parameters: time (1 mm), power (30W), and gas (air). Subsequently, cork particles undergo wet medium treatment with iron chlorides, maintaining a 2/1 molar ionic ratio of Fe^{3+}/Fe^{2+} at a solution pH of 2. The mixture is dropwise added to a pH 11 ammonium-dissociated solution, followed by neutralization and filtration. Then, the magnetic cork particles are introduced into a known Cr(VI) concentration solution. At defined intervals, cork particles are extracted, and 1,5-diphenylcarbazide is added, reacting with Cr(VI) and producing a distinct pinkish color. The resulting liquid is measured using ultraviolet-visible spectroscopy, compared with a pre-established calibration curve.

To complement this, cork particles are subjected to X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM). Findings reveal that within the initial 24 hours, Cr(VI) is effectively removed due to the collaborative reducing effects of cork and magnetite. The particles show the presence of Cr, Fe, C and O (Figure 1), in the analysis by EDAX. This Cr is Cr(III) and Cr metal when the study is carried out by XPS.

Keywords: cork, magnetic cork, Cr(VI), plasma treatment, adsorption.



Element	Weight %	Atomic %
C K	32.8	52.6
O K	28.2	33.9
Cr K	1.0	0.4
Fe K	38.0	13.1

Figure 1: EDAX APEX by SEM of magnetic cork particle after Cr(VI adsorption.

References:

1. Pereira, H. (1988), Chemical composition and variability of cork form Quercus suber L. *Wood SciTechnol* 22(3), 211–218.
2. Abenojar, J., Barbosa, A.Q., Ballesteros, Y., del Real, J.C., da Silva, L.F.M., Martínez, M.A. (2014), Effect of surface treatments on cork: surface energy, adhesion and acoustic insulation. *Wood Sci Technol* 48/1, 207-224.
3. Abenojar, J., López de Armentia, S., Barbosa, A.Q., Martínez, M.A., Velasco, F., da Silva, L.F.M., del Real J.C. (2020), Coating cork particles with iron oxide: effect on magnetic properties. *Wood Sci Technol* 54/1 869–889.

Improvement of Hydrophilicity of Composite Membrane and Its Application for Desalination

P. Rattana-arporn[1], S. Poompradub[1,2,3,*]
[1]Department of Chemical Technology, Chulalongkorn University, Bangkok, Thailand
[2]Center of Excellent in Green Materials for Industrial Application, Chulalongkorn University, Bangkok, Thailand
[3]Center of Excellence on Petrochemical and Materials Technology, Chulalongkorn University, Bangkok, Thailand

Abstract:

In this research, the hydrophilic polyethersulfone (PES)/silica composite membrane was developed via a sol-gel reaction. Firstly, the PES membrane was prepared by using a phase inversion. Two types of PES membranes (porous and dense membranes) were obtained under the different evaporation temperatures. The morphology of PES membrane was determined by using scanning electron microscopy (SEM). The create of more hydrophilic membrane is a common strategy to reduce membrane fouling during the desalination. By using the sol-gel technique, the in situ silica particles generated in PES membrane are of use to clarify not only the reinforcement effect but also the increased hydrophilicity of PES membrane to improve the efficiency of desalination. The advantage of this technique was the mild conditions, low cost, and low toxicity in aqueous systems and made a good silica dispersion. The effect of in situ silica content generated in PES membrane on the hydrophilicity, mechanical properties, permeate flux and salt rejection was investigated. It was found that the high water purity with salt rejection of 98.9 % was obtained by using the PES composite membrane prepared under evaporation temperature at 60°C with 7 wt% in situ silica content. Accordingly, the developed hydrophilic PES composite membrane in this research has the potential for desalination to produce clean water.

Keywords: hydrophilicity, in situ silica, membrane, sol-gel reaction, polyethersulfone, desalination

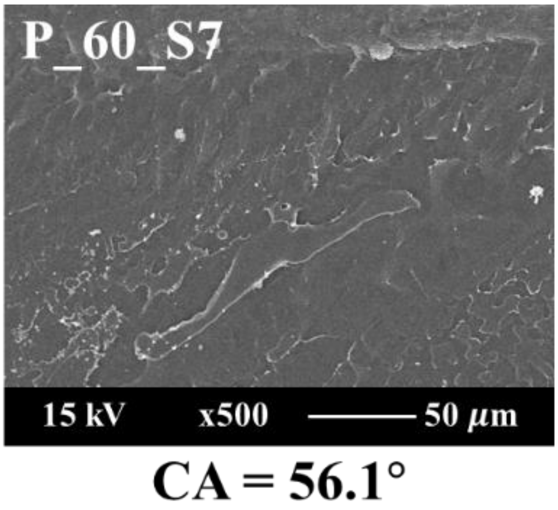


Figure 1: Cross-sectional SEM images of hydrophilic PES composite membrane by in situ sol-gel technique.

References:

1. Saleem, H., Trabzon, L., Kilic, A., Zaidi, S. (2020) Recent advances in nanofibrous membranes: Production and applications in water treatment and desalination, *Desalination*. 478, 114478.
2. Rastegarpanah, A., Mortaheb, H. (2016) Surface treatment of polyethersulfone membranes for applying in water treatment by direct contact membrane distillation, *J. Membr. Sci.*, 531, 99-107.

N_2/Ar dual-gas microplasma device conducting first-in-human wound treatment clinical trial

Shin-Chen Pan[1,2], Yu-Han Wei[3], Ming-Hsien Cheng[3], Jiunn-Der Liao[3,*]
[1] Division of Plastic and Reconstructive Surgery, Department of Surgery, National Cheng Kung University Hospital, College of Medicine, National Cheng Kung University, Tainan, Taiwan
[2] International Center for Wound Repair and Regeneration, National Cheng Kung University
Tainan, Taiwan
[3] Department of Materials Science and Engineering, National Cheng Kung University
Tainan, Taiwan
*Corresponding to: 9108026@gs.ncku.edu.tw)

Abstract

The Gentle Plasma Skin Regenerator Model 2 (GPSR_M2) is a non-thermal microplasma device (**Figure 1 (a)**) containing a 99% Ar/1% N_2 dual gas (i.e., Ar is used to excite the plasma and N_2 is used to enrich the plasma species and adjust the plasma temperature.). The plasma temperature is adjusted to about 40°C, while the plasma-wound working distance is 5 mm. GPSR_M2 has been undergone rigorous preclinical evaluation, including plasma treatment studies of *in vitro* and *in vivo* cellular wound healing mechanisms [1, 2], as well as experiments on small and extensive large animal wounds. [3, 4] GPSR_M2 is primarily designed to promote wound healing in humans, particularly in chronic cases such as diabetic foot ulcers (DFU), and therefore requires the submission of clinical trial data for its specific indications.

This first-in-human (FIH) study using GPSR_M2 to treat chronic DFU-related wounds has been approved by the National Cheng Kung University Hospital Institutional Review Board (NCKUH IRB) and the Taiwan Food and Drug Administration (TFDA). This clinical trial is a randomized, single-blind, two-group parallel study, including a plasma treatment group and a placebo (pure gas-only) group [5]. Wound area reduction serves as the primary endpoint, and the secondary endpoint includes wound infection extent and wound bacterial analysis. The trial requires a total of 68 patients (the first 4 clinical cases are shown in **Figure 1(b)~(d)**).

Safety assessment depends on the results of successive reviews by the ethics committee and TFDA. Results from these phases affirm the safety of the GPSR_M2 clinical trial, paving the way for the elevation of the FIH study on September 14, 2023. Throughout the trial, annual implementation reports will be submitted to the NCKUH IRB, supplemented by semi-annual reports to the TFDA. The dual agency approval underscores our commitment to review the efficacy of non-thermal microplasma in the treatment of diabetic foot ulcers.

Non-thermal microplasma has demonstrated its ability to stimulate cell proliferation and accelerate wound healing in diabetic mice and pigs, according to preclinical studies. The FIH study has so far involved 6 clinical cases, and the preliminary results further predict the effectiveness of GPSR_M2 non-thermal microplasma in promoting wound healing when treating diabetic foot ulcers.

Keywords: Non-thermal microplasma device, preclinical evaluation, diabetic foot ulcers, first-in-human study, wound area reduction, wound infection.

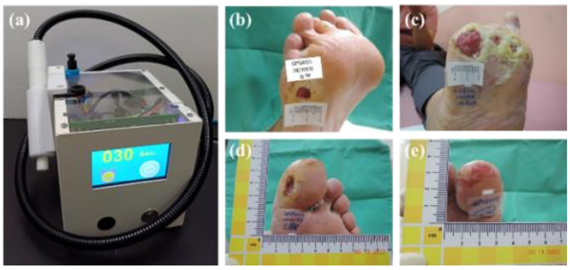


Figure 1: (a) is the non-thermal microplasma device GPSR_M2 used in clinical trials, (b) is the first case GPSR001, (c) is the second case GPSR002, (d) is the third case GPSR003, (e) is the fourth case GPSR004. The wound photos were taken on the screening day.

References:

1. M. H. T. Ngo, J. D. Liao, P. L. Shao, C. C. Weng, and C. Y. Chang, “Increased fibroblast cell proliferation and migration using atmospheric N2/Ar microplasma for the stimulated release of fibroblast growth factor-7,” Plasma Processes and Polymers, 11, 80-88, 2014.
2. M. H. Ngo Thi, P. L. Shao, J. D. Liao, C. C. K. Lin, and H. K. Yip, “Enhancement of angiogenesis and epithelialization processes in mice with burn wounds through ROS/RNS

signals generated by non-thermal N2/Ar microplasma," Plasma Processes and Polymers, 11, 1076-1088, 2014.

3. P. L. Shao, J. D. Liao, T. W. Wong, Y. C. Wang, S. Leu, and H. K. Yip, "Enhancement of wound healing by non-thermal N2/Ar microplasma exposure in mice with fractional-CO2-laser-induced wounds," PloS one, 11, e0156699, 2016.
4. P. L. Shao, J. D. Liao, S. C. Wu, Y. H. Chen, and T. W. Wong, "Microplasma treatment versus negative pressure therapy for promoting wound healing in diabetic mice," International Journal of Molecular Sciences, 22, no. 19, 10266, 2021.
5. B. Stratmann, T.-C. Costea, C. Nolte, J. Hiller, J. Schmidt, J. Reindel, K. Masur, W. Motz, J. Timm, and W. Kerner, "Effect of cold atmospheric plasma therapy vs standard therapy placebo on wound healing in patients with diabetic foot ulcers: a randomized clinical trial," JAMA network open, 3, no. 7, e2010411, 2020.

Acknowledgments:
This study is grateful for the support from the National Science Council Special Research Program (NSTC 111- 2811-E- 006- 011-MY3) and the National Cheng Kung University Hospital Affiliated Research Program (NCKUH-11202038).

Dielectric barrier discharge air plasma as a sustainable process for biorefinery development towards poly(3-hydroxybutyrate) production

C. Argeiti [1*], E. Stylianou[1], D. Ladakis[2], P.J Cullen[3], A. Koutinas[1]
[1] Department of Food Science and Human Nutrition, Agricultural University of Athens, Athens, Greece
[2] Department of Agricultural Development, Agri-Food and Natural Resources Management, National and Kapodistrian University of Athens, Athens, Greece
[3] School of Chemical and Biomolecular Engineering, University of Sydney, Sydney, Australia

Abstract:

Dielectric barrier discharge (DBD) air plasma can be used as a novel technique for sustainable biorefinery development of various industrial side streams.[1] Brewers' spent grains (BSG) contain a high amount of dextrins and other components, such as lignin, cellulose and hemicellulose. The polysaccharides and dextrins can be converted into fermentable sugars via enzymatic hydrolysis and used as carbon source for the production of value-added products through microbial fermentations. BDB air plasma can be used as an alternative to the convential methods for efficient lignocellulosic pretreatment in order to enchance the performance of the enzymatic hydrolysis. In this way ambient temperature and pressure is used, as well as dry gases instead of chemicals and solvents. In this study, DBD air plasma pretreatment of BSG was evaluated at different voltage values (100 V, 150 V, 200 V), treatment durations (10 min, 30 min, 60 min, 120 min), duty cycles (5%, 10%, 15%) and solids concentrations (50 g/L, 100 g/L, 125 g/L, 150 g/L, 175 g/L). The highest overall conversion yield of polysaccharides to sugars was obtained in the case of 150 g/L solids concentration at 10% duty cycle, 200 V, 1000 Hz and 30 minutes duration, reaching up to 73%. The produced hydrolysate after plasma treatment was used as carbon and nutrient source for the production of poly(3-hydroxybutyrate) (PHB). The results showed that when plasma treated hydrolysate was used as carbon and nutrient source during fermentation, the highest PHB concentration (74.4 g/L) was reached at an earlier stage compared to fermentations that BSG hydrolysate pretreated with convencial methods was used. The yield of the fermentation was 0.32 g/g and the productivity was 2.64 g/L/h.

Keywords: DBD air plasma, brewers' spent grains, biorefinery development, enzymatic hydrolysis, poly(3-hydroxybutyrate)

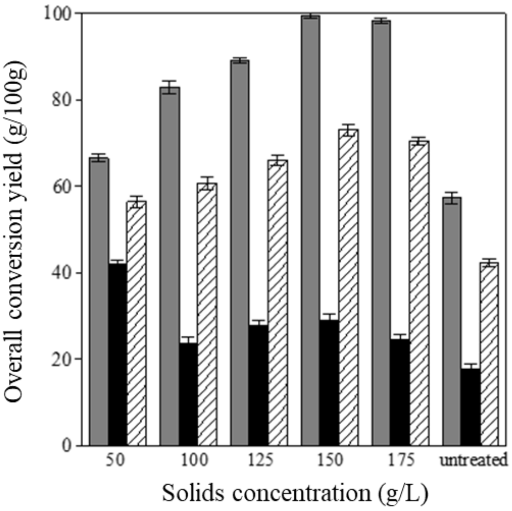


Figure 1: Overall conversion yield of glucan (grey bars), hemicellulose (black bars) and glucan/hemicellulose (white bars with lines) using the whole BSG suspension (solid and liquid fractions) derived after DBD plasma treatment. Different initial BSG concentrations (50 g/L, 100 g/L, 125 g/L, 150 g/L and 175 g/L) were used in DBD plasma treatment. Enzymatic hydrolysis was carried out at 100 g/L. DBD plasma treatment was carried out at 200 V, 30 min pretreatment duration, 1000 Hz discharge frequency and 10% duty cycle

References:

1. Vanneste, J., Ennaert, T., Vanhulsel, A., & Sels, B. (2017). Unconventional pretreatment of lignocellulose with low-temperature plasma. ChemSusChem, 10(1), 14-31.

Biorefinery electrification by dielectric barrier discharge plasma of lignocellulosic biomass in a semi-pilot microbubble reactor

E. Georgiadou, N. Giannakis, K. Filippi, A. Koutinas
Agricultural University of Athens, Department of Food Science and Human Nutrition,
Athens, Greece

Abstract:

Valorization of industrial side streams in the biorefinery context requires cost-effective and environmentally friendly pretreatment methods, to achieve lignin removal and high polysaccharide to sugar conversion yields (Wright et al., 2018). This study proposes electrification through plasma technology as pretreatment method of suspended brewers' spent grain (BSG). The production of reactive species in plasma activated water (PAW) at different working volume (300 mL, 1 L, 5 L, 10 L), duty cycle (5%, 12.5%, 20%) and air flow rate (1 mL/min, 3.3 mL/min, 6 mL/min) was evaluated. Fluctuation of pH, temperature and conductivity was characterised, while nitrates, nitrites, ozone and hydrogen peroxide were quantified in the generated plasma activated water. We observed that the lower the volume, the highest the final value for conductivity, temperature and concentration of nitrates and nitrites. In addition to this, discharge frequence was a critical parameter with a higher impact factor regarding its effect on conductivity, pH drop and nitrate production, compared to the duty step. While the increase in the duty cycle positively affected the properties of PAW, there were no significant differences observed by the increase of aeration. Water activation at 2600 Hz, 100 ds and 3.3 mL/min was the optimum condition regarding the production of reactive species in the 12 L reactor. This condition was selected for the plasma treatment of BSG and resulted in 97.6% glucan conversion and 54.5% hemicellulose conversion after 3.5 h, which is 33% and 41.2% higher than the control sample (Figure 1). This result indicates that plasma pretreatment of lignocellulosic biomass can be achieved at 10 L scale demonstrating that scale up of biomass pretreatment with DBD plasma is feasible. The production of sugar-rich hydrolysates could be enanced using only renewable electricity, avoiding the use of high temepratures and pressures, alkaline or acidic solutions and solvents.

Keywords: Brewers' spent grain, dielectric barrier discharge air plasma, reactive species, scale-up microbubble reactor

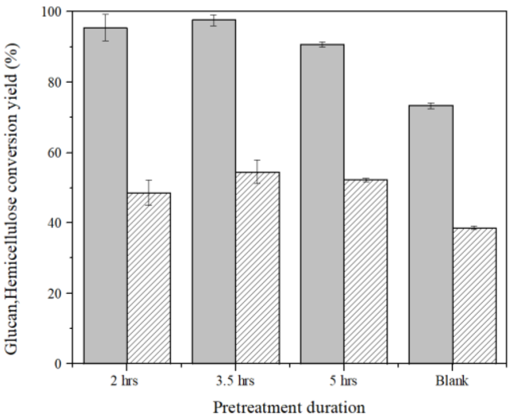


Figure 1: Overall conversion yileds of glucan (grey bar) and hemicellulose (crossed-line bar) after plasma treatment of BSG in 12 L microbubble reactor at 2600 Hz, 100 Dc, 3.3 vvm, 250 V at different pretreatment durations.

References:

1.Wright, A., Bandulasena, H., Ibenegbu, C., Leak, D., Holmes, T., Zimmerman, W., Shaw, A., Iza, F., 2018. Dielectric barrier discharge plasma microbubble reactor for pretreatment of lignocellulosic biomass. AIChE J. 64, 3803. https://doi.org/10.1002/AIC.16212

Radiative transfer in air-H_2O mixtures for LIBS applications: calculation of the Net Emission Coefficients and the Mean Absorption Coefficients

BENDIDA Imane [1,2*], CRESSAULT Yann [1], LIANI Bachir[2], VALENSI Flavien[1]
[1]LAPLACE, University of Toulouse , CNRS, INPT, UPS, Toulouse, France
[2] LPT, University of Tlemcen, Tlemcen, Algeria

Abstract:

As part of the problem of detecting pollutants in water using the LIBS (Laser Induced Breakdown Spectroscopy) technique, a small plasma is generated by focusing a laser pulse on a solid, liquid or gaseous sample of interest. Spectral analysis and numerical simulation of the light emitted by the plasma enable the atomic elements contained in the vaporised medium to be identified and quantified. LIBS is a technique with great potential, especially in the environmental field, and it is proving highly effective for characterising complex media without using traditional chemical treatments. Its versatility, ease of use, speed of response, sensitivity and the possibility of carrying out remote and in situ analyses mean that pollutants and their concentrations can be identified at any time, without risk.

Solving the radiative transfer equation is a fundamental approach to determining the radiative flux, absorption and emission within a plasma, a term in the bamance energy equation. Solving this equation directly using numerical methods such as finite element, finite difference or Monte Carlo methods is difficult because of the dependence on both spectral and geometric quantities, and is also very costly in terms of calculation time. The calculation of radiative transfer is therefore based on approximate methods defined as a function of geometric or spectral assumptions. These are the Net Emission Coefficient (NEC) and the P1 method using the MeanAbsorption Coefficients (MACs)

The NEC is the difference between the radiative power emitted in the axis of the arc and the radiation absorbed in other regions of the plasma by a unit of volume, in which it is assumed that the plasma is spherical, homogeneous and isothermal. This calculation is performed under atmospheric pressure in the temperature range of 300K to 30000K, for different plasma thicknesses between 0 and 100mm and under the assumption of local thermodynamic equilibrium (Figure 1). The results highlight the influence of three parameters on the net emission coefficients: temperature, pressure and plasma thickness. The values of NEC allow the estimations of the radiative losses in the hot regions of the plasma.Through spectral simplification, the P1 method uses the MACs. This method divides the radiative spretum into a given number of spectral intervals in which the total absorption is assumed to be constant. For an air-H_2O plasma, seven frequency intervals covering a wide spectral range were studied. The influence of temperature and pressure on the MACswas analysed by considering five different averages: classical, Planck, modified Planck, Rosseland and hybrid Planck-Rosseland. These MACs describe the absorption of radiation in the warm regions of the plasma.

Keywords: air-H_2O mixtures, Radiative Transfer Equation, equilibrium, Net Emission Coefficient, P1 method using Mean Absorption Coefficients.

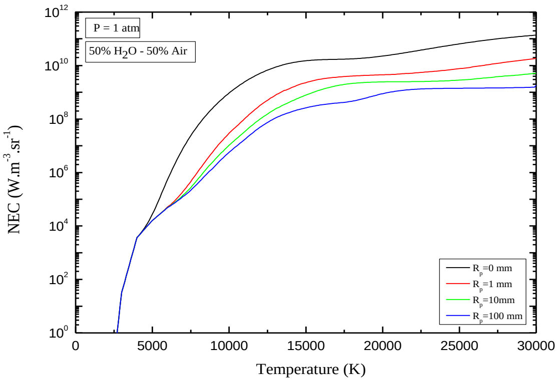


Figure 1: Total net emission coefficient of a 50% H_2O - 50% Air mixture: influence of plasma radius.

References:

1. Lowke.J.J., (1974) Prediction of arc temperature profiles ising approximate emission coefficients for radiation losses, *J.Quant. Specrosc. Radial. Transfer*,14, 111-112
2. Bartlova.M., Bogatyreva.N, and Aubrecht. V., (2014) Radiation heat transfer in thermal argon plasma with iron vapours, *Plasma Phys Technol*, 1, 8–10

Simultaneous production of hydrogen gas and carbon black nanoparticles using liquid phase plasma method

S.-C. Jung [1*], K.-H An [2]
[1] Dept. of Environmental Engineering, Sunchon National University, Sunchon 57922, Korea
[2] Dept. of Nano & Advanced Materials Engineering, Jeonju University, Jeonju 55069, Korea

Abstract:
In this study, carbon black particles were produced together with hydrogen from a benzene solution using the LPP process. For the LPP method to be superior to other hydrogen production methods, it is very important to secure economic feasibility by increasing the value of carbon black particles. Carbon black produced by the LPP method was heat-treated at various temperatures and applied as a conductive material for supercapacitors, and its performance was evaluated by comparing it with commercially available Super-P. When a plasma field is formed in a benzene solution using LPP, primary carbon black particles are generated together with hydrogen, and are agglomerated by solid-liquid separation and heat treatment to increase the particle size to a secondary structure. Carbon black was crystallized by heat treatment, and polygonal multiple nanoshells were formed by the formation of multi-graphenes. Also, as the heat treatment temperature increased, the pore volume and diameter increased, but the interlayer spacing $d_{(002)}$ decreased. The electrical conductivity of carbon black heat-treated at 1500 ℃ was the highest, and rather decreased when the temperature increased above that. The electrode used as the conductive material after heat treatment at 1500 ℃ showed the largest specific capacitance value and the smallest IR drop. Carbon black heat treated at 1,500 °C and 2,000 °C showed excellent charge and discharge characteristics of Super-P, which was thought to be due to its excellent conductivity and large specific surface area. The energy efficiency of the hydrogen production method by the LPP method performed in this study was evaluated to be superior to that of the water electrolysis method. In conclusion, since the carbon black produced by the LPP method showed better conductive properties than Super-P, it was possible to confirm the possibility as a new hydrogen production method.

Keywords: carbon black, Hydrogen production, lithium secondary batteries, supercapacitor, conductive agent, liquid phase plasma

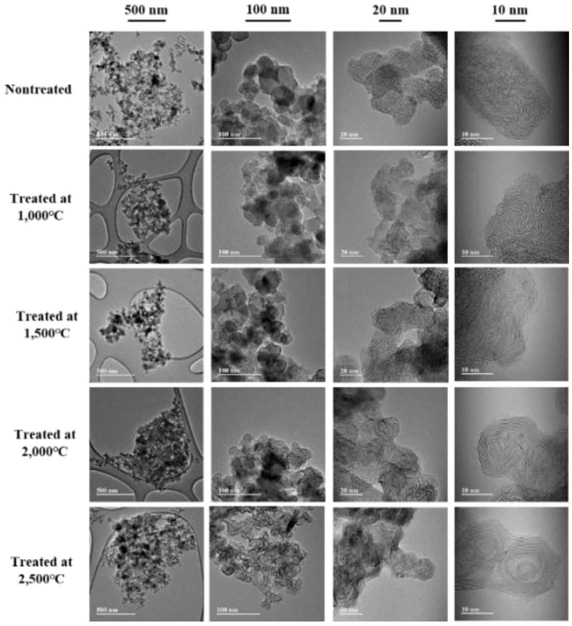


Figure 1: TEM images according to the plasma discharge time of carbon black particles produced from benzene, and TEM image of carbon black particles according to the heat treatment at each temperature.

References:

1. Raab, M., Korner, R., Dietrich, R.-U. (2022), Techno-economic assessment of renewable hydrogen production and the influence of grid participation, *Int. J. Hydrog. Energy* 47, 26798-26811.
2. Jeong, S., Chung, K.-H., Lee, H., Park, H., Jeon, K.-J., Park, Y.-K., Jung, S.-C., (2017), Enhancement of Hydrogen Evolution from Water Photocatalysis Using Liquid Phase Plasma on Metal Oxide-Loaded Photocatalysts, *ACS Sustainable Chem. Eng*. 5, 3659-3666.
3. Chung, K.-H., Lam, S.S., Park, Y.-K., Kim, S.-J., Jung, S.-C., (2022), Application of liquid-phase plasma for the production of hydrogen and carbon from the plasma-induced cracking of liquid hydrocarbons, *Fuel* 328, 125297.

Catalytic effects of the load-induced confinement within metallic interfaces on molecular dissociation

M. Vezzelli [1], E. Marquis [1], M.C. Righi [1]
[1] Department of Physics and Astronomy “Augusto Righi”, University of Bologna, Bologna, Italy

Abstract:

There is a growing consensus that both mechanical forces and confinement can enhance the chemical reactivity of molecules [1]. These aspects are strictly connected within the field of tribology, since decomposition of stable molecules is typically observed at the interface of mechanical components that are sliding under pressure. Tribochemical reactions are so inevitable that they are often exploited to promote the formation of tribofilm, which controls friction and wear during the movement of mechanical parts. These phenomena are not easily monitored by in situ experiments, while they can be investigated by *in silico* simulations. Here, we aim to rationalize the load-induced reactivity of confined molecules through density functional theory (DFT) simulations. In particular, fragmentation of molecules confined within metallic interfaces is systematically studied by applying a straightforward procedure that we named “Distance Sweep Analysis” (DSA). This analysis allows to induce confinement by gradually increasing the amount of external pressure acting on the surfaces, thus introducing mechanical energy into the system (Figure 1). After a pressure threshold is reached, the uncontrolled molecular fragmentation is observed, coinciding with the release of the accumulated mechanical tension. We studied the behavior of simple molecules such as H_2O and CH_4, confined within metallic interfaces of Fe(110) Ni(111) and Rh(111). Our results suggest that molecular confinement within metallic interfaces can lower the activation barriers for the dissociation. This behavior is correlated with the increased electron-charge accumulation at the interface gap, leading to greater charge redistributions that can popolate the antibonding orbitals of the molecule [2]. Although our preliminary results point towards highly system-dependent behavior, some general trends emerged. We believe that the analysis presented here can pave the way for a deeper understanding of mechano-confined reactivity in tribological systems.

Keywords: mechano-catalysis, tribo-chemistry, confinement, molecular dissociation, DFT.

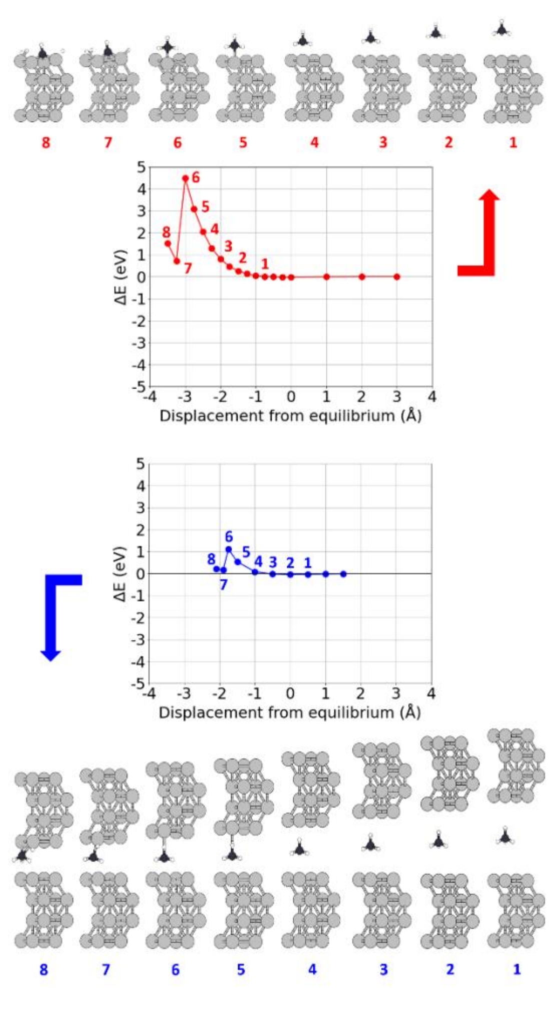


Figure 1: “Distance Sweep Analysis” (DSA) procedure, in which a CH_4 molecule is placed over a Rh(111) surface (above) and between the interface gap (below), showing the progressive confinement and the associated energy profile.

References:

1. Grommet, A. B.; Feller, M.; Klajn, R. Chemical reactivity under nanoconfinement. Nature nanotechnology 2020, 15, 256–271.
2. Martinez de la Hoz, J. M.; Balbuena, P. B. Small-molecule activation driven by confinement effects. ACS Catalysis 2015, 5, 215–224.

Ni-Al_2O_3 Cold Spray Coating: Effect of process parameters on Microstructure and Performance of the coating

Raihan A. Goriya [1,2,*], A.A. Sorour [1,2], Nasirudeen Ogunlakin [1]
[1] Interdisciplinary Research Center for Advanced Materials (IRS-AM), King Fahd University of Petroleum & Minerals, Dhahran, Kingdom of Saudi Arabia
[2] Mechanical Engineering Department, King Fahd University of Petroleum & Minerals, Dhahran, Kingdom of Saudi Arabia
*Corresponding Author: g202113450@kfupm.edu.sa

Abstract:

Cold spray coating technology has gained prominence in additive manufacturing, proving valuable for fabricating, restoring, and maintaining individual components. The deposition process plays a crucial role in preserving the inherent characteristics of the original phase while mitigating potential influences related to the surface coating and substrate material. Examining the impact of variations in process parameters on the coating characteristics of Ni-Al_2O_3 when cold sprayed onto 304 Stainless Steel is crucial. Utilizing computational tools, the goal is to identify optimized process parameters, minimizing the need for excessive experimental trials. A key aspect of this study involves a detailed microstructural analysis of the coated sample subjected to the cold spraying process. The investigation reveals a direct correlation between adjustments in process parameters and subsequent alterations in the coating's microstructure. These identified variations, illuminated by the microstructural analysis, hold significant implications for the mechanical properties of the cold spray coating. This research contributes to a nuanced understanding of the interplay between process parameters as shown in Table 1, microstructure, and mechanical properties in cold spray coating on 304 Stainless Steel, offering valuable insights for optimizing Ni-Al_2O_3 coatings and enhancing the overall performance of cold spray technology in additive manufacturing. In addition to the comprehensive analysis of coating characteristics, the study extends to adhesion testing—a critical aspect of evaluating coating performance. Three samples (T200P7, T400P7, and T600P7) underwent adhesion testing using a scratch tester equipped with a Rockwell C indenter featuring a 100 µm tip diameter. The indenter moved linearly at 5 mm/minute, with the applied load gradually increasing from 0.03 to 30 N at a rate of 15 N/min, covering a distance of 10 mm. The evaluation included determining critical loads, assessing adhesion strength, and identifying failure mechanisms. This holistic approach involved integrating acoustic emission, penetration depth analysis, and scrutinizing scanning electron microscopy (SEM) and optical micrographs of the scratch track. By incorporating adhesion testing, a comprehensive understanding of the mechanical characteristics and durability of Ni-Al_2O_3 cold spray coating on 304 Stainless Steel is sought, contributing to a more thorough comprehension of the coating's behavior in practical applications.

Keywords: cold spray coating, optimization, Ni-Al_2O_3, 304 stainless steel, process parameters, microstructural analysis

Table 1 Spray parameters of cold spray coating

Spray Parameters			
Designation	**T200P7**	**T400P7**	**T600P7**
Gas preheat temperature (°C)	200	400	600
Gas Pressure (Mpa)	0.7	0.7	0.7
Stand-off distance (mm)	15	15	15
Step Size (mm)	2	2	2
Traverse gun speed (mm/s)	30	30	30

Abrasion of Polyester Sportswear Materials: The Impact of Yarn Parameters and Aging

G. Čubrić [1], I. Salopek Čubrić[2], A. Petrov[1*]
[1] University of Zagreb Faculty of Textile Technology, Department of Clothing Technology, Prilaz baruna Filipovića 28 a, Zagreb, Croatia
[2] University of Zagreb Faculty of Textile Technology, Department of Textile Design and Management, Prilaz baruna Filipovića 28 a, Zagreb, Croatia

Abstract:

In the world of sports equipment, especially in the production of sportswear, the challenge of maintaining the durability of materials is becoming increasingly crucial to ensure performance and lasting quality. This scientific paper explores a set of materials intended for the production of football jerseys, all made of 100% polyester (both standard and recycled). The focus of the research is on the resistance of materials to abrasion. The resistance to abrasion was investigated for non-used materials, as well as for the materials that were aged. The aging of materials was in the form of the exposure to sunlight for 80 hours, simulating conditions during football games. The key phase of the research is centered on material abrasion before and after aging that is investigated using the AquaAbrasion tester. Through the simulation of 2500 and 5000 cycles of abrasion, the aim was to determine the durability of the materials and how their properties change due to use and aging. Related to it, additionally are observed the changes in the structure density, mass per unit area, and thickness. Special attention is given to recycled polyester material, exploring its sustainability and impact on quality compared to other materials. The results of this research provide valuable insights into the practical application of these materials in the production of sportswear and potential steps toward improving their durability and sustainability.

Keywords: abrasion, durability, yarn, sportswear, football, recycled, polyester, aging, sustainability.

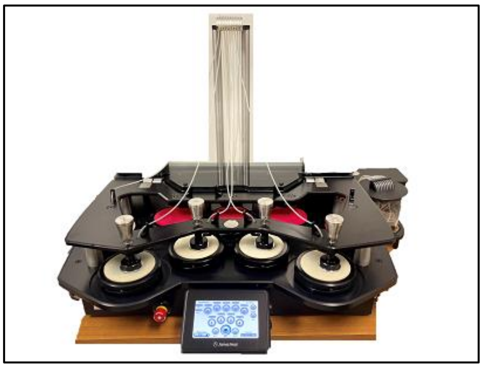


Figure 1: The image illustrates a modern abrasion simulation device named AquAbrasion, manufactured by the renowned company James Heal. This innovative equipment is specifically designed to simulate the abrasion of sportswear and active wear. In this paper, it is used to investigate the abrasion of materials intended for production of football jerseys, providing a comprehensive simulation of real-world usage conditions for thorough testing and analysis.

References:

1. Faheem, A., Khurram Shehzad, A., Wardah, A., Bushra, M., Abher, R., Sheraz, A., Farooq, A., Yasir, N. (2023) Recent Developments in Materials and Manufacturing Techniques Used for Sports Textiles, *Int. J. Polym. Sci.*, 1-20.
2. Toktas, S., Salopek Čubrić, I., Petrov, A. (2023) Influence of simulated outdoor wear on the performance of sportswear for a professional football player, Proceedings of the 9th International Ergonomics Conference, Lecture Notes in Networks and Systems. Salopek Čubrić I, Čubrić G, Jambrošić K, Jurčevič Lulić T, Sumpor D. (eds.). Cham: Springer Nature, 333-341.
3. Petrov, A., Salopek Čubrić, I., Čubrić, G., Katić Križmančić, I. (2022) Assessing the Impact of Outdoor Aging and Test Settings to Abrasion of Materials, 22th International Conference on Materials, Tribology & Recycling - MATRIB 2022. Bušić M, Leder Horina J, Tropša V. (eds.). Zagreb: Croatian society for materials and tribology, 330-344.

Effect of ceramic tribo-elements on tribological properties of rough steel surfaces

A. Dzierwa [1,*], R. Reizer [2]
[1] Faculty of Mechanical Engineering and Aeronautics, Department of Manufacturing Processes and Production Engineering, Rzeszow University of Technology, Rzeszow, Poland
[2] Institute of Materials Engineering, College of Natural Sciences, University of Rzeszow, Poland

Abstract:

Any new technical object constructed in accordance with the requirements contained in the design and technological documentation has its own, full operational potential. During operation this potential is decreased, in the result of the occurring physicochemical changes of elements, i.e. the wear of friction pairs, material fatigue, corrosive processes, etc. Destruction processes occur in the result of working conditions, referred to as wear [1], which cause a sudden or gradual loss of performance of the elements. Because wear in most cases leads to a reduction in the operating potential of machines and their components it should be countered. This countermeasures should start at the design stage, with appropriate selection of elements of the tribomechanical assembly, so as to reduce wear during operation. In addition to design, a number of technological methods of wear prevention are also applied. These can include, among others, the following:

- use of heat and thermo-chemical treatment (e.g. harden nitriding, carburizing, cyaniding),
- use of plastic forming of metals (e.g. hammering, burnishing),
- applying overlays and coatings (e.g. chemical nickel plating, hard facing).

Ceramics are used in a wide range of applications, both in lubricated and unlubricated contacts. The most common bulk ceramics in tribological use are based on alumina (Al_2O_3), silicon carbide (SiC), silicon nitride (Si_3N_4), zirconia (ZrO_2) or boron nitride (B_4N). To exemplify, silicon nitride may be used in bearings and metal cutting tools, alumina in cutting tools and hip joints, silicon carbide in mechanical seals, zirconia in dies and hip joints and boron nitride is mainly used in cutting and abrasive applications [2, 3].

Although many experimental works have been carried out on the relation between different ceramics and tribological properties of steel surfaces, influences of sliding velocity, normal load and kind of ceramic balls on the friction and wear property of steel-ceramics pairs have not yet been studied synthetically. Therefore, the comparative investigations on the tribological performance of the isotropic rough surfaces sliding against Si_3N_4, SiC, WC, Al_2O_3 and ZrO_2 as well as 100Cr6 balls will be deeply analyzed: Tribological tests were carried out on a T-11 pin/ball – disc tribological tester, in the ball - disc configuration. The tribological couple was a stationary ceramic ball and a disc made of 42CrMo4 steel with a hardness of 40±2 HRC. The finishing treatment of the discs was vapour blasting process (Figure 1). The sliding distance in all variants was 282 m. The tests were carried out at a load of 9.81 N and at three sliding speeds: 0.16; 0.32 and 0.48 m/s.

Keywords: surface topography, friction, wear, sand blasting.

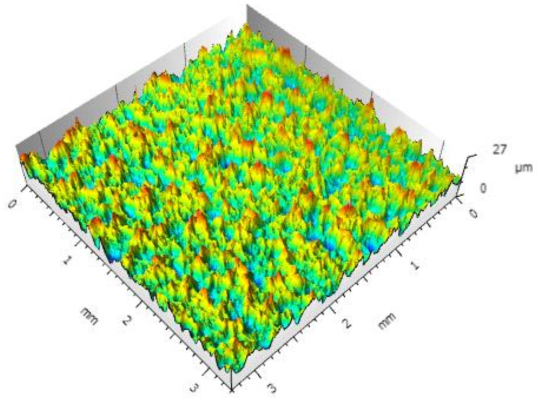


Figure 1: Isometric view of vapour blasted surface.

References:

1. Zaleski, K. (2017) The effect of vibratory and rotational shot peening and wear on fatigue life of steel, *Eksploatacja i Niezawodnosc – Maintenance and Reliability*, 19(1), 102-107.
2. Chen, M., Kato, K., Adachi, K. (2001), Friction and wear of self-mated SiC and Si3N4 sliding in water, *Wear*, 250(1-12), 246-255.
3. Andersson, P., Blomberg, A. (1993) Alumina in unlubricated sliding point, line and plane contacts, *Wear*, 170(2), 191-198.

Multi-criteria optimization of tribological properties of slide-burnished 36NiCrMo16 steel

A. Dzierwa [1,*], J. Sep [1], P. Pawlus [1]
[1] Faculty of Mechanical Engineering and Aeronautics, Department of Manufacturing Processes and Production Engineering, Rzeszow University of Technology, Rzeszow, Poland

Abstract:

Surface topography is one of the most important factors which affects the reliability and life of machine elements [1]. It has a structure composed of microscopic peaks and valleys formed during the manufacturing process and is widely present in various engineering practices [2]. Characteristics of tribological phenomena, like friction and wear, in dry or/and lubricated conditions, depend on areal surface topographies of sliding surfaces. In order to reduce friction and wear and improve reliability, various design and treatment methods have been applied consistently in mechanically interacting surfaces. One of such treatments is slide burnishing process (Figure 1). Therefore, the aim of this study was to examine the influence of slide burnishing process parameters (load, sliding distance, sliding speed) on tribological properties (coefficient of friction, wear, weight loss) using the Taguchi method. The multi-objective optimization method by The Technique for Order of Preference by Similarity to Ideal Solution (TOPSIS) technique has been applied to predict the best values of influencing parameters to obtain the optimum tribological characteristics.

Slide burnishing tests were conducted at room temperature using Haas CNC Vertical Mill Center VF-1. The samples to be burnished had a diameter of 25.4 mm and a height of 6 mm so that they could be used for tribological tests. Tribological tests were conducted using a tribological tester T-11 in a ball-on-disc configuration. The levels of tested input factors in tribological investigations were as follows: applied load: 5, 10 and 15 N, sliding speed: 0.24, 0.48 and 0.72 m/s, and sliding distance: 140, 280, and 420 m. Dry and lubricated sliding tests were carried out at room temperature (20–22 °C). Surface topography after slide burnishing process as well as after tribological tests, was measured by a white light interferometer Talysurf CCI Lite. After measurements, at least 7 profiles were taken in four positions (90^0 apart) perpendicular to the wear track in order to obtain the cross-sectional area of the wear tracks. Then, the values of the cross-sectional area of the wear track (in TalyMap software defined as the area of the hole) were averaged and the value of volumetric wear was calculated using Formula:

$$VD = \pi d S,$$

where:

d - diameter of the wear track,

S - area of the hole (the cross-sectional area of the wear track).

The worn-out samples were also analyzed using SEM to reveal the wear mechanism.

Keywords: friction, wear, slide burnishing, surface topography.

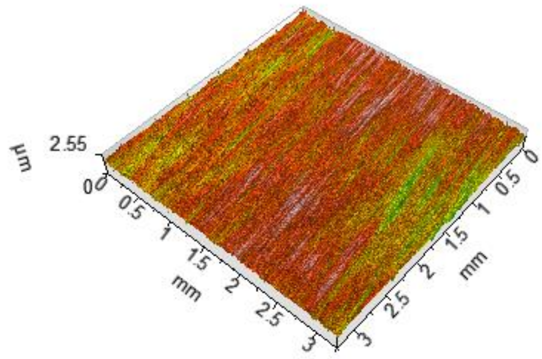


Figure 1: Isometric view of slide burnishing surface.

References:

1. Zhang, X., Pan, X., Wang, G. (2019) Influence factors of surface topography in micro-side milling, *Int. J. Adv. Manuf. Technol.*, 12, 5239-5245.
2. Shi, R., Wang, B., Yan, Z., Wang, Z., Dong L. (2019), Effect of surface topography parameters on friction and wear of random rough surface, *Materials*, 12, 2762.

Evaluation Method for Dynamic Damage Performance of Materials under High-Frequency Impact

M. Hua [1,2,*], X. Sui [2,3]

[1] Lanzhou Huahui Instrument Technology Co., Ltd., Lanzhou, China
[2] Lanzhou Institute of Chemical and Physics, Lanzhou, China
[3] Institute for Engineering Design and Product Development Tribology Research Division TU Wien, Vienna, Austria

Abstract:

High-frequency fatigue testing machines play a crucial role in the field of engineering, especially in evaluating the fatigue performance of components and materials. With the continuous development and application of modern engineering technology, the requirements for the fatigue performance of materials and components are increasing. Fatigue failure under impact is a common phenomenon in engineering, prevalent in equipment such as aerospace, weaponry, and forging machinery subjected to high-intensity impacts. It is also a significant cause of overload damage to mechanical components. To more accurately assess the fatigue behavior of materials and components under high-frequency impact, high-frequency fatigue testing machines have emerged. These machines are widely used in industries such as aerospace, automotive, electronics, and marine to evaluate the fatigue performance of materials and components in complex working environments. Particularly in the aerospace industry, there is an urgent demand for such testing machines.

This article designs a high-frequency fatigue testing device, which operates as follows: the main shaft is driven by a motor, and power is transmitted to the horizontal shaft through a transmission shaft. The horizontal shaft then transfers the power to the lifting mechanism of the impact wheel, enabling vertical reciprocating motion of the specimen table. The lifting mechanism of the impact wheel moves vertically upward to impart impact on the specimen, and after completion, it moves vertically downward to complete the reset function. This process is repeated to achieve high-frequency impact on the specimen by the pressure head. The periodic contact between the specimen table and the loading pressure head in the lifting mechanism of the impact wheel realizes impact fatigue. Precise impact is achieved by adjusting the loading force in the loading mechanism and setting the lifting height of the lifting mechanism. Additionally, high-speed rotation of the impact block achieves impact frequencies exceeding 2Hz.

Keywords: Fatigue performance, Impact testing machine, Surface engineering, Multifunction.

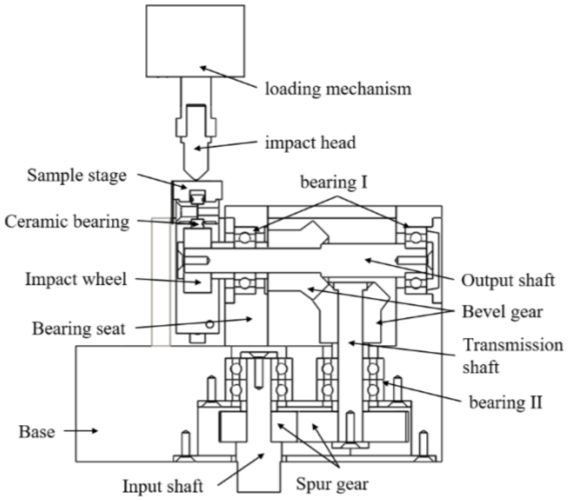


Figure 1: Figure illustrating the schematic diagram of instrument mechanical principles and structure.

References:

1. Kawamoto M , Yasuda T , Tanaka T ,et al.On a New Testing Machine for Repeated Impact under High Frequency[J].journal of the Japan Society for Testing Materials, 1961, 10(88):35-39.
2. Liu X D , He J G , Fu G Y ,et al.MCU-Based Driven Design for the Electromagnetic Valve of the Repeated Impact Testing Machine[J].Applied Mechanics & Materials, 2011, 43:317-320.
3. Xu M , Fu G , Liu L ,et al.A melioration of the new type of repeated impact testing machine[J].Journal of Suzhou University(Engineering Science Edition), 2009.

Protic ionic liquids as additives in water-based ecolubricants

P. Mostaza-Ucedo[1*,] J. Arias[1], M.D. Avilés[1] and F.J. Carrion-Vilches[1]

[1]Grupo de Ciencia de Materiales e Ingeniería Metalúrgica. Universidad Politécnica de Cartagena
C/ Doctor Fleming sn, 30201 Cartagena (SPAIN)

Abstract:

Ionic Liquids have demonstrated their ability as lubricants, or as additives for lubricants. Due to their intrinsic properties such as their broad liquid range from as low as -100ºC to as high as 350ºC, they have practically no vapor pressure, and exhibit high thermal stability and decomposition temperatures above 300ºC. Their high polarity induces self-assembly films on metallic surfaces, which enable the ionic liquid to prevent direct contact and reduce friction and wear of the surfaces in contact [1-3].

Protic ionic liquids (PIL) are synthesized in a cleaner and simpler way than conventional ionic liquids and have only organic elements in their composition. They have emerged as being excellent additives to formulate water-based lubricants due to their excellent solubility, low or non-toxic effect and high biodegradability, amongst other properties. [4-6].

In this work, two PIL, bis(2-hydroxy ethyl) ammonium caprylate (DC8) and undecylenate (DC11), are studied as additives at 1, 2, 5, and 10 wt.% in water-based lubricants, in a pin-on-disc configuration using the tribopair sapphire/ stainless steel. The friction coefficients and wear rates data are discussed as a function of the critical micelle concentration of the PIL in the aqueous solution (Figure 1). In addition, the Stribeck curves were studied to display the lubrication regimes, whilst techniques such as XPS, Raman spectroscopy, or electronic microscopy were used to establish the friction and wear mechanisms present in the tribological contacts.

Keywords: protic ionic liquid, water-based lubricant, tribology, friction coefficient, wear rate.

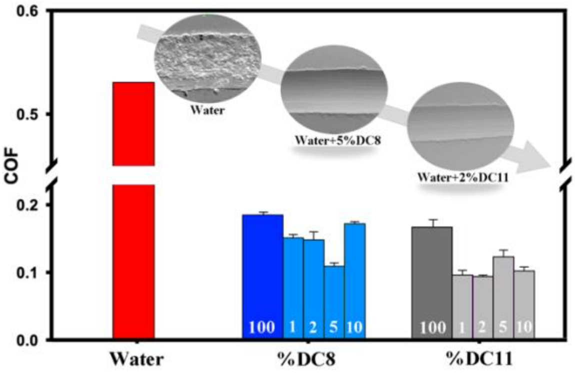


Figure 1: Friction coefficients of the protic ionic liquids DC8 and DC11 water-based lubricants at 1, 2, 5, and 10 wt.%.

References:

1. M.D. Bermúdez, A.E. Jiménez, J. Sanes, F.J. Carrión. (2008), Ionic liquids as advanced lubricant fluids. Molecules 14, 2888-2908
2. I. Minami. (2009), Ionic Liquids in Tribology. *Molecules* 14, 2286-2305.
3. P. Calandra, E.I. Szerb, D. Lombardo, V. Algieri, A. De Nino and L. Maiuolo. (2021), A presentation of ionic liquids as lubricants: some critical comments. *Appl. Sci.* 115677.
4. M. D. Avilés, F. J. Carrión, J. Sanes, and M. D. Bermúdez. (2018), Effects of protic ionic liquid crystal additives on the water-lubricated sliding wear and friction of sapphire against stainless steel. *Wear* 408, 56–64.
5. F. J. Carrión, M. D. Avilés, K. Nakano, C. Tadokoro, T. Nagamine, and M. D. Bermúdez. (2018), Diprotic ammonium palmitate ionic liquid crystal and nanodiamonds in aqueous lubrication. Film thickness and influence of sliding speed. *Wear* 418–419, 241–252.
6. T. Espinosa, M. Jiménez, J. Sanes, A. E. Jiménez, M. Iglesias, and M. D. Bermúdez. (2014), Ultra-low friction with a protic ionic liquid boundary film at the water-lubricated sapphire-stainless steel interface. *Tribology Letters* 53, 1–9.

Influence of Tribo-Films of Calcium Detergents on Micro-Pitting Behaviour of Steels

A. Tada [1,2,3,*], D. Spaltmann [2], K. Tagawa [3], V. L. Popov [1]
[1] Institute of Mechanics, Technical University of Berlin, Berlin, Germany
[2] Tribologie und Verschleißschutz,
Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany
[3] Lubricants R&D Dept., ENEOS Corporation, Yokohama, Japan

Abstract:

Electrified vehicles attract much attention to achieve carbon neutrality via reducing carbon dioxide emissions. For increase of their range, one promising method is the reduction of transmission torque loss by lowering viscosity of lubricants in transaxles [1]. However, this generally reduces oil film thicknesses and thus leads to severe lubrication conditions. As a consequence, sometimes fatigue is caused, such as micro-pitting at gear contacts. While various lubricant additives which form tribo-films on metal surfaces are known to affect formation of micro-pits [2], the influence of calcium detergents is not well investigated. To clarify their influence, slip-rolling tests were carried out in this study, using twin disc tribo-meters. The tribological testing conditions were set so that they were like those at gear contacts (the oil temperature: 100 °C, slide-to-roll ratio: -9%, the maximum Hertzian contact pressure: 2 GPa). Two kinds of discs made from SCM420H steel were used: specimen discs and counter discs. Counter discs had larger roughness and higher hardness (R_a: 0.3 µm, HRC: 61) than those of specimen discs (R_a: 0.04 µm, HRC: 58) to promote micro-pitting generation on specimen discs. The tests were carried out up to 7 million cycles, and the disc surfaces were observed with optical and confocal microscopes by stopping the tests at certain cycles. Four kinds of lubricants were formulated to investigate the effect of calcium detergents (Table 1). By comparing Oil A, B. and C, it was found that adding tricresyl phosphate and Ca sulfonates lead to reduction of wear but accelerated micro-pit generation (Figure 1). On the other hand, Ca salicylates (Oil D) did not increase micro-pit area, reducing wear at the same time. In addition, the degree of micro-pitting was correlated to roughness reduction of counter discs. Oil D showed larger roughness reduction than Oil C, indicating that enough running-in process which prevented micro-pitting acceleration was obtained with Ca salicylates. This difference might be related to the formation rate of tribo-films. To clarify the mechanisms, further analyses on the tribo-films are currently proceeded.

Table 1: Formulation of lubricants

Oil	A	B	C	D
PAO	Bal.	Bal.	Bal.	Bal.
AO, mass%	0.5	0.5	0.5	0.5
TCP, massppm(P)		800	800	800
Ca Sul., massppm (Ca)			600	
Ca Sal., massppm (Ca)				600

PAO: Poly-α-olefin, AO: antioxidant
TCP: Tricresyl phosphate
Sul.: Sulfonates, Sal.: Salicylates

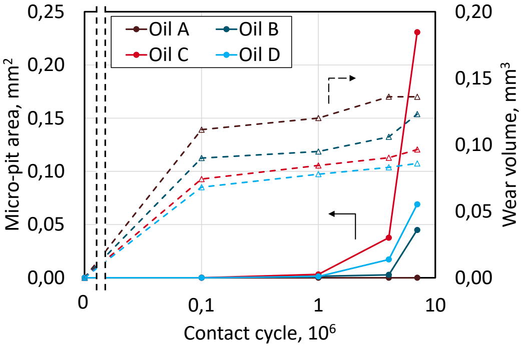


Figure 1: Wear volume and micro-pit area of specimen discs.

Keywords: tribology, micro-pitting, wear, tribo-film, lubricants, additives, calcium detergents.

Acknowledgements:

The authors thank ENEOS for the financial support, C. Opitz (BAM), Y. Kanno, M. Okamura, S. Hasegawa and Central Research Laboratory (ENEOS) for their experimental supports.

References:

1. Tada, A. et al., (2023) Development of Transaxle Fluid for Electrification Vehicles: Design of Novel Additive Formulation, *SAE Int. J. Adv. & Curr. Prac. in Mobility* 5(3) 1055-1062.
2. Ueda, M. et al, (2019) *In-situ* observations of the effect of the ZDDP tribofilm growth on micropitting, *Tribol. Int.* 138, 342-352.

SETCOR
Conferences & Exhibitions

SETCOR
Conferences & Exhibitions