HIPIMS 2023

13th International Conference on HIPIMS

Thirteenth International Conference on HIPIMS

12-15 June 2023, Venlo, The Netherlands

Sheffield Hallam University · National HIPIMS Technology Centre UK · Fraunhofer IST · Network of Competence INPLAS · SEA Surface Engineering Association · SELF Surface Engineering Leadership Forum · SVC Society of Vacuum Coaters · Henry Royce Institute · Catapult High Value Manufacturing · IOM3 Institute of Materials, Minerals & Mining

Foreword

On behalf of the Organising committee, I am pleased to welcome you to the 13th International Conference on HIPIMS! Looking back in time, we fondly remember the HIPIMS Days hosted at Sheffield Hallam University in the UK in 2004. Focusing on the novel HIPIMS technology, the event was the first of its kind and, after 6 years, it became the progenitor of the conference we know today. Building on the success of the HIPIMS Days, the International Conference on HIPIMS was created in partnership with Fraunhofer IST in Germany, and was endorsed by the European Joint Committee on Plasma and Ion Surface Engineering.

The HIPIMS technology has now irrefutably taken its place amongst the highly successful plasma-based manufacturing technologies worldwide. Every year new organisations from around the world are welcomed into the dynamic family of HIPIMS researchers. In the UK, the National HIPIMS Technology Centre strives to contribute to society through its collaboration with the High Value Manufacturing Catapult - one of the largest catalysts for future growth and success of manufacturing in the UK, and has the support of the Henry Royce Institute and the Surface Engineering Association to further strengthen the impact of HIPIMS for Industry and Academia.

An important highlight of this year's Conference is the "Quo Vadis Surface Engineering" panel discussion on the topic of "Surface Engineering for the Hydrogen Economy. Opportunities and challenges: the industry perspective" chaired by David Elliott , CEO of the Surface Engineering Association, UK. An exciting collection of research presentations underpins the theme, covering fundamental processes, materials and applications alike. The conference brings together new advances in understanding of HIPIMS plasmas and thin film growth and innovative applications in fields as wide ranging as hard coatings to semiconductors.

Another special aspect of the conference is that it coincides with the 40th anniversary of IHI Hauzer Techno Coating - one of the leading PVD/PACVD coating equipment suppliers. Hauzer was one of the companies who acquired a commercial licence to practice surface pre-treatment using HIPIMS at the earliest stages of the technology's evolution. Indeed, the first industrial coating system in the world enabled with HIPIMS technology was a Hauzer HTC1000-4 coater, which was put into operation in 2010 at Sheffield Hallam University. The National HIPIMS Technology Centre, UK in collaboration with Ionbond UK and supported by Hauzer engineers has recently introduced the first in UK Research Platform for Digitalisation of PVD Processes including HIPIMS on Industrial Scale using a Hauzer HTC 1000-4 platform. It is my pleasure to congratulate the company for its longevity and innovative spirit.

I would also like to take the opportunity to deeply acknowledge the long-standing, generous and unconditional support of our sponsors: IHI Hauzer Techno Coating, IHI Ionbond, Trumpf Hüttinger and CemeCon.

Finally, a special note of gratitude is extended to the dedicated team of volunteers at Hauzer, Sheffield Hallam University, SEA and Fraunhofer IST who, over the past year, have contributed remarkable efforts and enthusiasm in making this meeting a success.

I hope you will enjoy the meeting and come away inspired, invigorated and infused with new ideas.

Prof. Arutiun P. Ehiasarian
Head of the National HIPIMS Technology Centre, UK
Conference Chairman

Foreword by Hauzer

On behalf of Hauzer, as well, I'd like to welcome you to this 13th International Conference on HIPIMS, held in the Netherlands as part of our 40th anniversary celebration. We're proud and pleased to be able to host the conference again this year, after a successful event for Hauzer's 25th anniversary in 2008.

This was made possible thanks to the long-standing relationship between Hauzer and Sheffield Hallam University (SHU), which started when Wolf-Dieter Münz, then working at Hauzer, joined SHU in 1993 to establish the Chair for Surface Engineering. Since then, several Hauzer employees have received their MSc or PhD degrees from SHU, and this is true for Hauzer's sister corporation Ionbond as well. Over the decades, we've been happy to continue this close relationship with such an innovative institute, now through our connection to Papken Hovsepian and Arutiun Ehiasarian. We are looking forward to seeing how they will continue to break new ground in the exciting field of HIPIMS.

Thanks to the energy transition and digitisation, the traditional markets for hard coatings such as automotive, tool and decorative industry are going through tremendous changes. Just think of the shift to e-mobility. These changes will have a massive impact on the global industry. At the same time, this creates new opportunities for growth and new markets, applications and technologies to develop. Societal changes are always a call to keep innovating – and this is also true for surface technologies.

At Hauzer, we believe strong partnerships are crucial for this innovation. Now, and in the future, all of us need to work towards a world that is much less dependent on fossil fuels. To achieve this, conferences like this one remain critical to exchange the latest research results and trend information. This collaborative and pioneering approach is part of the Hauzer DNA since it was started 40 years ago. It's helped us to grow and become the global leader in PVD equipment we are now – and aim to continue being.

We hope you enjoy the International Conference on HIPIMS as much as we will,
and we're looking forward to a bright future together.

Dave Doerwald
CEO at IHI Hauzer Techno Coating B.V.

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HIPIMS 2023

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Technical Programme

Thirteenth International Conference on HIPIMS

12-15 June 2023, Venlo, The Netherlands

DAY ONE | Wednesday 14 June 2023

8:00-8:10 am

Opening address by Prof. Arutiun P. Ehiasarian, *National HIPIMS Technology Centre, UK*, Prof. Dr. Christoph Herrmann, *Fraunhofer IST, Germany*, Dr. Dave Doerwald, CEO, *Hauzer, The Netherlands.*

First Morning session

Session Chairman: Prof. Arutiun P. Ehiasarian, *National HIPIMS Technology Centre, UK, Sheffield Hallam University, UK*

	Presentation time	Questions & Answers
1. Quo Vadis SE talk: **D. Elliott,** *SEA/SELF, UK* **"Surface Engineering, the key to a sustainable future"**	8:10 - 8:30	
2. **Christoph Herrmann**, Sabrina Zellmer, Ralf Bandorf, *Fraunhofer Institute for Surface Engineering and Thin Films IST, Germany* **"The Hydrogen Campus Salzgitter an Innovative Hub for Transformation of the Region"**	8:30 - 8:45	8:45 - 8:50
3. **Lucia Mendizabal**, Antía Villamayor, Eva G-Berasategui, *TEKNIKER, Spain* **"High volume magnetron sputtering: time and cost effective technology to manufacture critical components in PEMWE"**	8:50 - 9:05	9:05 - 9:10
4. **Ton Hurkmans**, *IHI Ionbond Group, The Netherlands* **"Ionbond's DOT's technology (thermal spray!) on titanium bipolar plates for electrolyzers"**	9:10 - 9:25	9:25 - 9:30
Coffee break	9:30 - 9:45	

Second Morning session

Session Chairman: Dr. Ralf Bandorf, *Fraunhofer IST, Germany*

	Presentation time	Questions & Answers
5. **Chongchong Tang**[1], Michael Dürrschnabel[1], Ute Jäntsch[1], Michael Klimenkov[1], Martin Steinbrück[1], Sven Ulrich[1], Marcus Hans[2], Jochen M. Schneider[2], Michael Stüber[1], [1)]*Institute for Applied Materials (IAM-AWP), Karlsruhe Institute of Technology (KIT), Germany,* [2)]*Materials Chemistry, RWTH Aachen University,Germany* **"Solid Solution and Ordered Quaternary MAX Phase Thin Films: Phase Formation and Reaction Mechanisms in Magnetron Sputtered Nanostructured Multilayers"**	9:45 - 10:00	10:00 - 10:05
6. **Jochen M. Schneider**, Materials Chemistry, *RWTH Aachen University, Germany* **"Thin film materials design & some thoughts on complexity and sustainability"**	10:05 - 10:20	10:20 - 10:25
7. **Vincent Ott**, Tomasz Wojcik, Christian Schäfer, Sven Ulrich, Szilard Koloszvari, Peter Polcik, Paul Mayrhofer, Helmut Riedel, Christoph Pauly, Michael Stüber, *Institute for Applied Materials, Karlsruher Institut für Technologie, Germany* **"Novel approaches for the PVD synthesis of advanced aluminide thin films: The example of Ruthenium-Aluminide"**	10:25 - 10:40	10:40 - 10:45
Coffee break	10:45 - 11:00	

Third Morning session

Session Chairman: Prof. Jaroslav Vlcek, *University of West Bohemia, Czech Republic*

	Presentation time	Questions & Answers
8. **Louise Antoine**[1], Aurélie Achille[2], Angéline Poulon-Quintin[2], Dominique Michau[2], Marjorie Cavarroc[1], *[1)]SAFRAN Paris-Saclay – SAFRAN Tech, F-78772 Magny-Les-Hameaux, France* **“Physical properties of pure tantalum nitride coatings”**	11:00 - 11:15	11:15 - 11:20
9. M-P. Besland[1], N. Ginot[2], T. Minea[3], A. Caillard[4], J. Zgheib[1], A. Rhallabi[1], **P-Y. Jouan**[1], *[1)]Université de Nantes, CNRS, Institut des Matériaux Jean Rouxel, IMN, France,* *[2)]Université de Nantes, CNRS, Institut d’Electronique et des Technologies du numéRique, France, [3)]Université Paris Saclay, CNRS, Laboratoire de Physique des Gaz et Plasmas, France, [4)]Université d’Orléans, CNRS, Groupe de Recherche sur l’Energétique des Milieux Ionisés, France* **“Improvement of Crystallinity and Deposition Rate Using Multi e-HiPIMS“**	11:20 - 11:35	11:35 - 11:40
10. **Pauline Kümmerl**, Sameer Aman Salman, Marcus Hans, Daniel Primetzhofer, Peter Polcik, Szilard Kolozsvari, Jochen M. Schneider, *Materials Chemistry (MCh), Lehrstuhl für Werkstoffchemie, RWTH Aachen, Germany* **“Oxidation behaviour of HIPIMS and DC magnetron sputtered (Hf1-xAlx) by thin films”**	11:40 - 11:55	11:55 - 12:00
Conference photograph and lunch break	12:00 - 13:00	

First Afternoon session

Session Chairman: Prof. Günter Bräuer, *TU Braunschweig, Germany*

	Presentation time	Questions & Answers
11. **Ryan Bower**[1], Jiaze Sun[1], Daniel D. Price[1], Bruno Rente[1], Ethan Muir[2], Papken Eh. Hovsepian[2], Arutiun P. Ehiasarian[2], R. Oulton[1], Peter K. Petrov[1], *[1)]Imperial College London, UK, [2)]National HIPIMS Technology Centre, UK, Sheffield Hallam University, UK* **“Preparation of robust nanostructured antimicrobial surfaces from TiN thin films deposited via HIPIMS”**	13:00 - 13:15	13:15 - 13:20
12. Christian Poltorak[1], Andreas Bergmaier[2], Klaus Seemann[1], Michael Stüber[1], **Sven Ulrich**[1], *[1)]Karlsruhe Institute of Technology (KIT) - Institute for Applied Materials (IAM-AWP), Germany, [2)]Universität der Bundeswehr München, Institut für Angewandte Physik und Messtechnik (LRT2), Germany* **“Constitution and properties of TiC1-x:H/a-C:H nanocomposite thin films prepared by HiPIMS processes at low and elevated temperature“**	13:20 - 13:35	13:35 - 13:40
13. **M. Kaufman**, J. Vlcek, J. Houska, S. Farrukh, *University of West Bohemia, Czech Republic* **“Low-temperature reactive HiPIMS of high-performance thermochromic VO2-based coatings for energy-saving smart windows”**	13:40- 13:55	13:55-14:00
Coffee break	14:00 - 14:15	

Second Afternoon session

Session Chairman: Dr. Chris Constable, *Ionbond, UK Ltd., UK*

	Presentation time	Questions & Answers
14. **Philipp Immich**, Louis Tegelaers, Geert-Jan Fransen, Huub Vercoulen, Gabriela Negrea, Ruud Jacobs, Andreas Fuchs, Daniel Barnholt, *IHI Hauzer Techno Coating B.V., The Netherlands* **"HIPIMS - The tool for a modern production for coat tools and components"**	14:15 - 14:30	14:30 - 14:35
15. **Jan Philipp Liebig**, Veit Schier, *Walter AG, Germany* **"Al-rich (Al,Ti)N at the cutting edge: Insights into the incipient nucleation of the B4 phase"**	14:35 - 14:50	14:50 - 14:55
16. **Stephan Bolz**, Biljana Mesic, Werner Kölker, Oliver Lemmer, *CemeCon, Germany* **"The Influence of Pulse Parameters on Plasma Properties and Performance of HiPIMS Coatings for Cutting Tools"**	14:55 - 15:10	15:10 - 15:15
17. **Maurizio Giorgio**[1], M. Steinhorst[1,2], S. Topalski[1], C. Leyens[1,2], T. Roch[1], *[1)]Fraunhofer Institute for Material and Beam Technology IWS, Germany, [2)]Institute of Materials Science, Technische Universität, Germany* **"Performance and perspectives of roll-to-roll coatings for metallic bipolar plates"**	15:15 - 15:30	15:30 - 15:35
Coffee break	15:35 - 15:50	

Third Afternoon session

Session Chairman: Prof. Jochen Schneider, *Materials Chemistry, RWTH Aachen University, Germany*

	Presentation time	Questions & Answers
18. **Kristian A. Reck**[1], Yusuf Bulut[2,3], Thomas Strunskus[1], Jonas Drewes[1], Peter Muller-Buschbaum[3], Stephan V. Roth[2,4], Franz Faupel[1], *[1)]Faculty of Engineering at Kiel University, Germany, [2)]Deutsches Elektronen-Synchrotron (DESY), Germany, [3)]Lehrstuhl fur Funktionelle Materialien, Technische Universit at Munchen (TUM), Germany, [4)]KTH Royal Institute of Technology, Department of Fibre and Polymer Technology, Germany* **"Real-time monitoring of early stages of HiPIMS film formation on polymers with GISAXS"**	15:50 - 16:05	16:05 - 16:10
19. **J. Vlcek**, *University of West Bohemia, Czech Republic* **"Deposition rate of the dielectric films prepared using reactive HiPIMS can be very high"**	16:10 - 16:25	16:25 - 16:30
20. **P. Zeman**[1], S. Haviar[1], M. Červená[1], A. Bondarev[2], R. Čerstvý[1], *[1)]Department of Physics and NTIS – European Centre of Excellence, University of West Bohemia, Czech Republic, [2)]Department of Control Engineering, Faculty of Electrical Engineering, Czech Technical University in Prague, Czech Republic* **"Dual-phase nanocomposite coatings based on crystalline ZrN and glassy ZrCu"**	16:30 - 16:45	16:45 - 16:50
21. **Holger Kersten**[1], Mathis Klette[1], Thomas Trottenberg[1], Sven Gauter[2], Maik Fröhlich[3], *[1)]Kiel University, Germany, [2)]Dyson, UK, [3)]FH Zwickau, Germany* **"Non-conventional plasma diagnostics – also for HiPIMS processes?"**	16:50 - 17:05	17:05 - 17:10
Coffee break	17:10 - 17:25	

Fourth Afternoon session

Session Chairman: Dave Elliott, *Surface Engineering Association, UK*

	Presentation time
Quo Vadis panel discussion session: **"Surface Engineering for Hydrogen Economy. Opportunities and challenges: the industry perspective"**	17:25 - 18:00
End of first day	18:00
Conference Dinner	19:00 - 21:30

DAY TWO | Thursday 15 June 2023

First Morning session

Session Chairman: Prof. Ludvik Martinu, *Polytechnique Montreal, Canada*

	Presentation time	Questions & Answers
22. K. Bobzin, C. Kalscheuer, M. P. Möbius, **C. Schulze**, *Surface Engineering Institute IOT, RWTH Aachen University, Germany* **“Prediction of OES data based on HPPMS process parameters by ANN”**	8:00 - 8:15	8:15 - 8:20
23. **Piotr Rozanski**, *TRUMPF Huettinger Sp. z o.o., Poland* **“Effect of metal oxide dielectric layers deposition by High Power Impulse Magnetron Sputtering on Resistive-Random Access Memory performance”**	8:20 - 8:35	8:35 - 8:40
24. **Stefan Körner**[1,2], Ralf Bandorf[2], Holger Gerdes[2], Thomas Schütte[3], G. Bräuer[1], *1)Institute for Surface Technology IOT (TU Braunschweig), Germany, 2)Fraunhofer Institute for Surface Engineering and Thin Films IST, Germany, 3)PLASUS GmbH, Germany* **“Reactive High Power Impulse Magnetron Sputtering (HIPIMS) of titanium oxide: Transition from metallic to poisoned regimes analysed by optical emission spectroscopy”**	8:40 - 8:55	8:55 - 9:00
Coffee break	9:00 - 9:15	

Second Morning session

Session Chairman: Dr. Ton Hurkmans, *IHI Ionbond Group, The Netherlands*

	Presentation time	Questions & Answers
25. **Christopher Fleming**[1], A. P. Ehiasarian[1], A. Sugumaran[1], P. Eh. Hovsepian[2], *1)Advanced Forming Research Centre, University of Strathclyde, Scotland, UK, 2)National HIPIMS Technology Centre, MERI, Sheffield Hallam University, UK* **“Extending the lifetime of H13 hot forging dies with a HIPIMS CrAlYN/CrN multilayer coating”**	9:15 - 9:30	9:30 - 9:35
26. T. Omiya[1,3], M. Fontes[1,2], T. Vuchkov[1,3], S. Cruz[1,3], A. Cavaleiro[1,3], **F. Ferreira**[1,3,4], *1)University of Coimbra, CEMMPRE Centre for Mechanical Engineering Materials and Processes, Department of Mechanical Engineering, Portugal, 2)Federal Institute of Education, Science and Technology of Sao Paulo, Brasil, 3)LED&Mat-IPN, Instituto Pedro Nunes, Laboratório de Ensaios Desgaste e Materiais, Portugal, 4)Walker Department of Mechanical Engineering, The University of Texas at Austin, USA* **“Tribological performance of doped-DLC coatings produced by HiPIMS in the presence of environmental-friendly additives”**	9:35 - 9:50	9:50 - 9:55
27. **João Carlos Oliveira**[1], Alireza Vahidi[1], Ricardo Serra[1], A. Cavaleiro[1,2], *1)Department of Mechanical Engineering, University of Coimbra, Portugal, 2)IPN – Instituto Pedro Nunes, Portugal* **“Carbon hybridization states quantification by EELS and NEXAFS in hard DLC films deposited by HiPIMS-DOMS”**	9:55 - 10:10	10:10 - 10:15
Coffee break	10:15 - 10:30	

Third Morning session

Session Chairman: Dr. Dave Doerwald, *IHI Hauzer Techno Coating B.V., The Netherlands*

	Presentation time	Questions & Answers
28. **Jörg Pantförder**, *iwis mobility systems GmbH & Co. KG, Germany* **"PVD coating in the production environment Industry 4.0"**	10:30 - 10:45	10:45 - 10:50
29. **Ralf Bandorf**[1], Holger Gerdes[1], Stefan Körner[1,2], Felix Huth[2], *[1)]Fraunhofer Institute for Surface Engineering and Thin Films IST, Germany, [2)]Technische Universität Braunschweig, Institut für Oberflächentechnik IOT, Germany* **"Reactive HIPIMS of Oxides for Industrial Processes"**	10:50 - 11:05	11:05 - 11:10
30. **Thomas Schütte**[1], Jan-Peter Urbach[1], Peter Neiß[1], Stefan Körner[2,3], Holger Gerdes[3], Ralf Bandorf[3], *[1)]PLASUS GmbH, Germany, [2)]Institute for Surface Technology IOT (TU Braunschweig), Germany, [3)]Fraunhofer Institute for Surface Engineering and Thin Films IST, Germany* **"Advanced Process Control for Metallic and Reactive HIPIMS Applications for Production Lines"**	11:10 - 11:25	11:25 - 11:30
Coffee break	11:30 - 11:45	

Fourth Morning session

Session Chairman: Holger Gerdes. *Fraunhofer IST, Germany*

	Presentation time	Questions & Answers
31. L.B. Varela-Jimenez[1], V. Simova[1], A. Miletic[1], O. Zabeida[1], P. Immich[2], G. Negrea[2], R. Schäfer[3], D. Schorn[5] Th. Schuette[4], S. Williams[6], J.E. Klemberg-Sapieha[1] and **L. Martinu**[1], *[1)]Department of Engineering Physics, Polytechnique Montreal, Canada, [2)]IHI Hauzer Techno Coating BV, The Netherlands, [3)]Robeko GmbH & Co. KG, Germany, [4)]MAGPULS GmbH, Germany, [5)]PLASUS GmbH, Germany, [6)]Sputtering Components Inc, USA* **"Tuning Residual Stress Depth Profile for Enhanced Tribological Properties of TiAlN Coatings Prepared by HiPIMS Using a Cylindrical Magnetron"**	11:45 - 12:00	12:00 - 12:05
32. **Ricardo Sera**, S.A. Adebayo, J.C. Oliveira, *CEMMPRE, Mechanical Engineering department, University of Coimbra, Portugal* **"Thick and smooth nanostructured Cr coatings by HiPIMS"**	12:05 - 12:20	12:20 - 12:25
33. **Chayan Ranjan Das**, Mufaddal Rangwala, Amitava Ghosh, *Indian Institute of Technology Madras, India* **"Influence of Ti and Si content on the structure and mechanical characteristics of HiPIMS deposited TiAlSiN nanocomposite"**	12:25 - 12:40	12:40 - 12:45
Lunch break	12:45 - 13:45	

First Afternoon session

Session Chairman: Dr. Philipp Immich, *IHI Hauzer Techno Coating B.V., The Netherlands*

	Presentation time	Questions & Answers
34. **A. Bertè**, P. Civardi, L. Parenti, P. Colombi, A. Sordi, *R&D Department, Lafer Rivestimenti Spa, Italy* **"Industrial scale innovative doping of DLC coatings deposited by MW-PACVD technique: mechanical and physical characterisation"**	13:45 - 14:00	14:00 - 14:05
35. Marek Betiuk[1], Aleksandra Mirońska[1], **Anna W. Oniszczuk[2]**, Wojciech Trzewiczyński[2], Wojciech Gajewski[2], Piotr Domanowski[3], *[1)]Łukasiewicz, Research Network, Institute of Precision Mechanics (IMP), Poland, [2)]TRUMPF Huettinger sp. z o.o. Zielonka, Poland,* *[3)]Department of Production Engineering, University of Technology and Life Sciences in Bydgoszcz, Poland* **"Comparative study of DC and HIPIMS discharge characteristics of Cr cylindrical magnetron"**	14:05 - 14:20	14:20 - 14:25
36. **H. Gerdes**, F. Oldenburg, A. Pflug, R. Bandorf, M. Vergöhl, C. Herrmann, *Fraunhofer IST, Germany* **"Digital Transformation in Thin Film Deposition"**	14:25 - 14:40	14:40 - 14:45
Coffee break	14:45 - 15:00	

Second Afternoon session

Session Chairman: Prof. Kirsten Bobzin, *IOT - Surface Engineering Institute, RWTH Aachen University, Germany*

	Presentation time	Questions & Answers
37. **Ivailo Dolchinkov,** Gerrit Jan van der Kolk, Ton Hurkmans, *IHI Ionbond AG, The Netherlands* **"Doped ta-C films deposited by pulsed Arc"**	15:00 - 15:15	15:15 - 15:20
38. **Mathis Klette[1]**, Martin Kopte[2], Wolfgang Fukarek[2], Holger Kersten[1], *[1)]Kiel University, Germany, [2)]VTD Vakuumtechnik Dresden GmbH, Germany* **"Characterization of a Pulsed Plasma and Macroparticles in an Industrial Scale ta-C Laser-Arc Coating System"**	15:20 - 15:35	15:35 - 15:40
39. J. **Vyskočil**[1], P. Mareš[1], M. Čada[2], Z. Hubička[2], J. Vlček[3], J. Blažek[1], T. Mates[1,4], I. Venkrbcová[4], *1)HVM PLASMA, Czech Republic,* *[2)]Institute of Physics, Czech Academy of Sciences, Czech Republic, [3)]HVM PLASMA, Czech Republic, [4)]Institute of Physics, Czech Academy of Sciences, Czech Republic* **"Properties of hard C coating prepared by different HIPIMS processes"**	15:40 - 15:55	15:55 - 16:00
Coffee break	16:00 - 16:15	

Third Afternoon session

Session Chairman: Dr. Chris Constable, *Ionbond, UK Ltd., UK*

	Presentation time	Questions & Answers
40. **Wojciech Trzewiczyński**[1], Anna W. Oniszczuk[1], Wojciech Gajewski[1], Jerzy Robert Sobiecki[2], *[1)]TRUMPF Huettinger sp. z o.o. Zielonka, Poland, [2)]Division of Surface Engineering, Warsaw University of Technology, Poland* **"Effect of current-voltage characteristic on plasma ionization in HIPIMS discharge of graphite target in argon atmosphere"**	16:15 - 16:30	16:30 - 16:35
41. **M. Učík**[1,2], J. Hnilica[1], P. Klein[1], P. Vašina[1], J. Klusoň[2], M. Jílek[2], A. Lümkemann[3], *[1)]Department of Physical Electronics, Masaryk University, Czech Republic, [2)]PLATIT a.s., Czech Republic, [3)]PLATIT AG, Switzerland* **"Novel Sputter Cathode Design to Obtain Very High Ionized Flux Fraction of Deposited Material in Industrial Conditions"**	16:35 - 16:50	16:50 - 16:55
42. **Szilárd Kolozsvári**, Peter Polcik, *Plansee Composite Materials GmbH, Germany* **"Target design for HIPMIS process challenges."**	16:55 - 17:10	17:10 - 17:15
43. **J. Capek**, K. Shaji, S. Haviar, *Department of Physics and NTIS - European Centre of Excellence, University of West Bohemia, Czech Republic* **"Preparation of multi-composite nanoparticle-based thin films for gas sensing"**	17:15 - 17:30	17:30 - 17:35
44. Olayinka O. Abegunde[1], Mohammed Makha[1], Karima Machkih[1], Hicham Larhlimi[1], **Anas Ghailane**[1,2], Youssef Samih[1], and Jones Alami[1], *[1)]Department of Materials Science, Energy and Nano-engineering, Mohammed VI Polytechnic University (UM6P), Morocco, [2)]Avaluxe Coating Technology, Germany* **"Structural, mechanical and corrosion resistance of Phosphorus-doped TiAlN thin film"**	17:35 - 17:50	17:50 - 17:55
Student Prize announcement, closing remarks: **A. P. Ehiasarian, R. Bandorf**	17:55 - 18:10	
End of conference	18:10	
Hauzer Dinner	19:00	

NOTES

HIPIMS 2023

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Abstracts

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12-15 June 2023, Venlo, The Netherlands

Physical properties of pure tantalum nitrides coatings

Transition metal nitrides coatings are widely studied because of their good optical, mechanical, thermal… properties. Depending on the stabilised crystalline structure, coatings present different properties. In particular, cubic and hexagonal tantalum nitride (TaN) coatings can be deposited. The aim of this study is to compare hexagonal and cubic-TaN electrical and adherence properties. Both phases were deposited by reactive high power impulse magnetron sputtering (HiPIMS) and reactive radiofrequency magnetron sputtering (RF-MS), on 316L stainless steel and AlN substrates. Characterisation of crystalline phases and films microstructure was realised by *Grazing* incidence X-ray diffraction (GIXRD) and scanning electron microscopy (SEM). Adherence was quantified by scratch tests for coating on steel substrate. Nanohardness measurement were realized on coating deposited on AlN substrates. Electrical properties were explored with a four-point probe on the previous samples. Grain morphology of RFMS coatings are columnar independently of the deposit phase (figure 1-a). HiPIMS technique leads to denser coating with nanograins (figure 1-b). In this study, we prove that a correlation exist between the process, the phase and the properties, for electrical and adherence. Explanations are proposed.

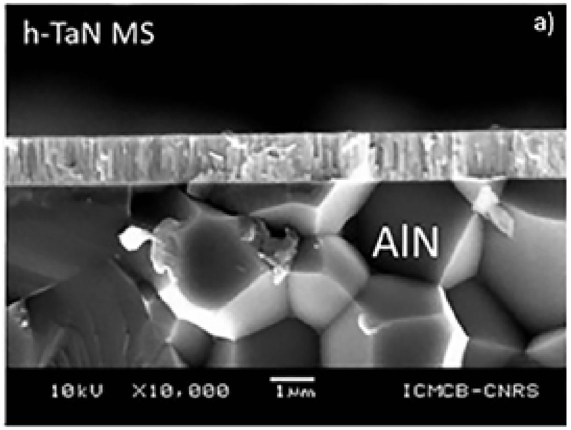

Fig 1-a: SEM pictures of cub-hex TaN HiPIMS

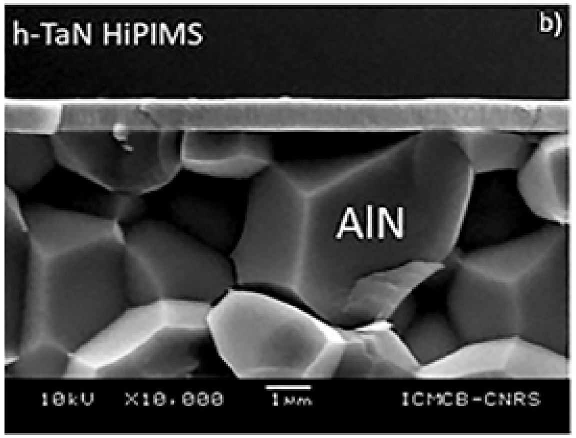

Fig 1-b: SEM pictures of hex-TaN HiPIMS

Louise Antoine[1]
Aurélie Achille[2]
Angéline Poulon-Quintin[2]
Dominique Michau[2]
Marjorie Cavarroc[1]

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Reactive HIPIMS of Oxides for Industrial Processes

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Most of the deposited thin films in magnetron sputtering are reactively deposited. This, of course applies as well for high power impulse magnetron sputtering (HIPIMS). As discussed in numerous papers, there are different effects in HIPIMS that lead to a vanishing (for small targets) or at least reduced hysteresis behaviour. Nevertheless, for industrial size cathodes and especially highly reactive materials like oxides a hysteresis occurs and needs to be handled.

This talk will emphasize different aspects of reactive HIPIMS on large scale cathodes. Different approached for active feedback control will be discusses, as well as aspects of the reactive gas inlet and design. Finally, different oxides from cylindrical cathodes with 500 mm and 750 mm, respectively will be presented.

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Industrial scale innovative doping of DLC coatings deposited by MW-PACVD technique: mechanical and physical characterisation

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The industrial scale DLCs coatings depositions are running with different PACVD techniques and gases species, all of them allowing the creation of a specific sp2 to sp3 ratio and, therefore, mechanical and physical properties. Nowadays, Diamond Like Carbon coatings are widely spread on a very high variety of applications, starting from engine related components and arriving to medical implants. Fundamental properties like high hardness, low COF and extremely low wear rate are at the base of the success of DLC. Nevertheless, there are possible applications where the high elastic modulus and hardness of traditional DLC are not matching with requirements; either a lower surface energy or even a higher corrosion resistance or electric conductivity can be of interest. To the author's knowledge, this is the first time that a PACVD industrial machine with microwave sources is used to modify the coating structure (Cr+W-C:H+a-C:H) with the addition of dopants for the a-C:H phase. In the first stage the origin formulation had been deposited and tested; following 3 different flows of Nitrogen had been investigated and, as last, a mixture of Nitrogen and Silicon.

Only the functional layer with formulation X-a-C:H had been affected by modifications.

From tests performed in Lafer's laboratory, there are several modifications to the coating properties by the addition of Nitrogen, as first. Even if keeping a relatively high hardness, indentation elastic modulus lowered and internal stresses, which we measured applying the Stoney equation on bendable strips. On the other hand, very low COF, lower than 0.1, on Pin-on-disc test had been measured, together with a good wear resistance, in the range of E-16 $m^3 N^{-1} m^{-1}$. Other relevant properties of the film changes with the dopants additions, starting from CIE colour coordinates and electric resistivity too. Surface energy values had been measured with contact angle technique and corrosion resistance in 3% NaCl water solution tested with a galvanostat apparatus.

The structures were investigated using an SEM with EDS, to explore changes in the film growth. A great number of interesting outcomes arrived from data. For sure, the addition of Nitrogen is giving DLC electric conductivity, increasing with Nitrogen flow. Also, the addition of Nitrogen is reducing internal stresses and reducing L* value, resulting in a deeper black coating. The combination of Silicon and Nitrogen results in a corrosion resistance improvement.

Fig: SEM SE fracture of N-doped DLC structure.

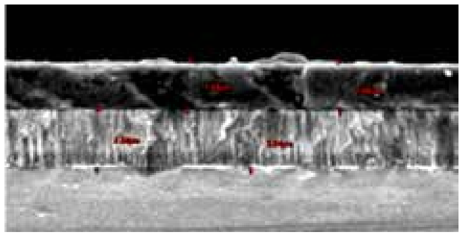
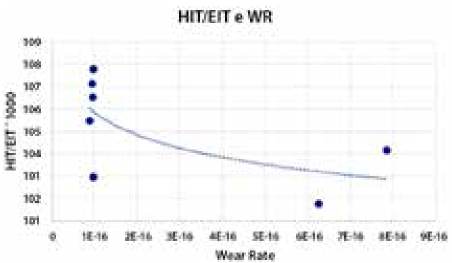


Fig: Point chart showing the modification of Hit/Eit ratio for the different formulations taken into exam.



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The Influence of Pulse Parameters on Plasma Properties and Performance of HiPIMS Coatings for Cutting Tools

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To increase energy efficiency in industries such as automotive and aircraft, the weight of modern materials must be reduced and their strength increased at the same time. The demands on coatings for cutting tools used for economical machining of such materials are therefore constantly increasing. One of the highest challenges in the cutting sector is machining of difficult-to-machine materials like stainless steel, hardened steel, titanium- and high-temperature alloys. Machining them requires high demands on adhesion, hardness, surface finish and chemical resistance of coatings. In this context, high-performance coating technologies, such as HiPIMS, play an increasingly important role. Thanks to HiPIMS dense, hard, adhesive and droplet-free layers can be deposited in highest quality with high energy efficiency at high deposition rates. The presentation shows the relationship between HiPIMS pulse parameters, plasma properties, coating properties and cutting performance of coated cutting tools. Thus, by tuning HiPIMS pulse parameters a significant increase in metal ionization could be detected by optical emission spectrometry, accompanied by improved coating properties of an (Al,Ti,Si)N layer. Finally, the improved coating properties led to a significant increase in machining performance, proven in turning of austenitic stainless steel.

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Preparation of robust nanostructured antimicrobial surfaces from TiN thin films deposited via HIPIMS.

Antimicrobial resistance is a growing issue in hospitals and public spaces, with high contact surfaces such as door handles, worktops and bedframes identified as major transmission points for bacteria and other pathogens. Surfaces coatings that offer antimicrobial resistance will help to lower the number of infections and minimise the use of antibiotics. A challenge is to make durable antimicrobial surfaces that will not lose their effectiveness over time. Nanostructured, ultrahard coatings deposited by HIPIMS are a potential route to achieve such surfaces.

Here, I will report the preparation and characterisation of some biomimetic antimicrobial surfaces consisting of TiN nanofeatures. TiN thin films were deposited via High Power Impulse Magnetron Sputtering onto steel and glass substrates at the National HIPIMS Technology Centre, Sheffield Hallam University and characterised at Imperial College London using XRD and spectroscopic ellipsometry. Colloidal lithography is then combined with Chemically Assisted Ion Beam Etching (CAIBE) to produce nanostructured surfaces consisting of TiN nanopillars and cones with varying pitch and aspect ratio. The nanostructured surfaces are characterised via SEM, AFM, and UV vis spectroscopy.

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Preparation of multi-composite nanoparticle-based thin films for gas sensing

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[1] S. Haviar et.al., International Journal of Hydrogen Energy, 43 (2018) 22756.

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Conductometric gas sensors based on metal-oxide semiconductors (MOS) are cheap, prompt, and stable devices. Their sensing mechanism is based on the modulation in electrical conductivity due to adsorption-desorption reactions between the target gas and the sensor surface.

A large surface area is desirable for gas sensing since this allows more interaction area for the target gas. Nanostructuring the sensing material thus increases the effective surface area thereby enhancing the sensorial response. MOS, both n-type and p-type, have shown remarkable gas sensing responses towards oxidizing and reducing gases which are infamous for being combustible or hazardous. In several cases, the combination of different MOS has been shown to synergistically improve the gas sensing performance via formation of highly sensitive heterojunctions at their interface.

Previous research at our laboratory has shown that n-type WO3 thin films decorated with p-type CuO nanoparticles (NP) resulted in promising sensorial response [1]. The enhanced response is attributed to the formation of nano p-n heterojunctions at the interface of the two MOS. Using this concept, we have prepared thin films composed of NPs of CuOx and WOx to realize a maximum number of nano p-n junctions.

In this work, the films were prepared using the Nanogen-Trio NP source equipped with three 1" magnetrons – Cu, W, Pd, mounted on a custom-built deposition chamber and sputtered by a DC power supply. However, due to the small target dimensions, all three targets exhibited a low sputtering rate in argon. The depositions were hence executed in Ar + O_2 gas mixture, where the oxygen flow rate giving the highest deposition rate was carefully chosen from the respective hysteresis curves. During deposition, the operation of each magnetron was controlled using an in-house developed software allowing preparation of a mixture of NPs with a defined volumetric ratio of the individual materials.

This work demonstrates the versatility of our custom-built gas aggregation source that enables effective control of the deposition parameters for each target material thereby facilitating the preparation of multi-composite NP-based thin films giving the best sensorial response.

Influence of Ti and Si content on the structure and mechanical characteristics of HiPIMS deposited TiAlSiN nano-composite

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In recent years, High Power Impulse Magnetron Sputtering (HiPIMS) PVD deposition technology has emerged as a superior alternative to direct current magnetron sputtering (DCMS) due to better coating properties, smoothness and substrate-coating adhesion [1]. High peak power with a small pulse duration is used in HiPIMS technology to achieve a much denser plasma in the order of 10^{18} m^{-3}[2], which results in dense, defect-free, and good quality smoother coating [3]. In the present investigation, TiAlSiN coatings have been deposited on WC-10Co (wt%) cermet with two different Ti-Si contents by state-of-the-art HiPIMS technology. Two types of targets TiSi23 and TiSi34, along with TiAl60 in common, were used for the deposition. The effect of Ti and Si at% on the microstructure and mechanical properties of the coatings was investigated by X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), 3D-surface profiling, nano-indentation, Rockwell indentation-adhesion, and scratch test. The thickness and deposition rate of the coating were estimated from the cross-section morphology. It could be inferred that the crystalline TiAlN phase was likely to be wrapped in the amorphous phase of Si_3N_4 in TiAlSiN coatings. With the increase of Ti atomic percentage in the coating, the hardness could be enhanced up to 42 GPa. In addition, coating surface roughness decreased from 14.3 nm to 10.9 nm, as shown in **Fig**, and grain size from 16.2 nm to 12.4 nm. The adhesion strength observed under the Rockwell-indentation test was of HF1 classes in both cases. Patterns of cracks, as observed under indentation test, were very similar to one another. However, a significant improvement in coating-substrate adhesion was realized with the increase of Ti content, as investigated through the Scratch test. The scratch test achieved a maximum load of 173 N during full delamination (LC_3) of TiAlSiN from the carbide substrate.

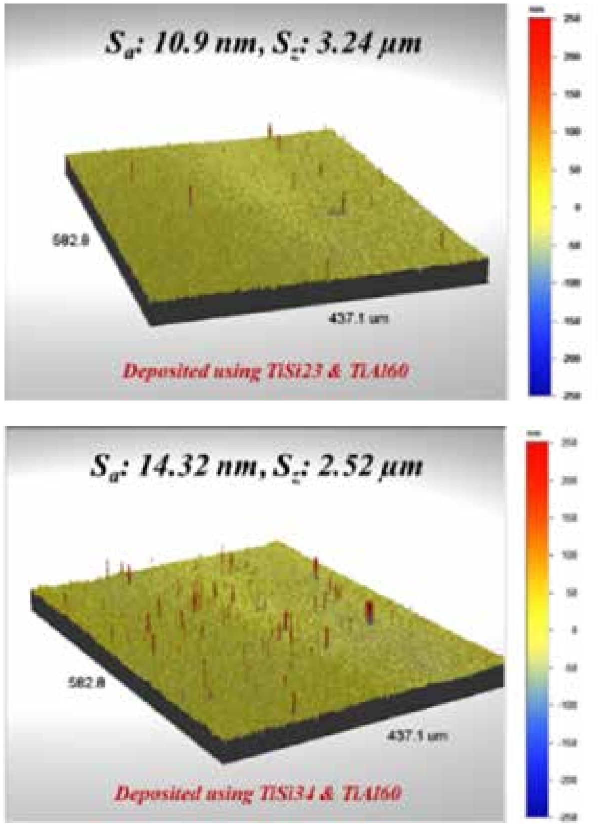

Fig: 3D surface morphology of the TiAlSiN coated surface deposited using two different target combination

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Doped ta-C films deposited by pulsed Arc

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Keywords:
Tetrahedral amorphous Carbon (*ta*-C); Pulsed arc deposition; doped ta-C, nano-structure

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Tungsten (W) doped and Boron (B) doped hydrogen-free tetrahedral bonded amorphous Carbon films (*ta*-C) were deposited using pulsed arc. The pulsed arc discharge has been operated with a very steep rise rate of the arc current reaching its maximum peak, upon which the pulse discharge is switched off. As a result, the plasma densities are very high.

During this time, the target surface undergoes changes that are different from a typical DC arc discharge. The present study shows how the target surface influences the deposition process and the coating properties of the deposited films. Coating properties are influenced by the target composition and structure as well, i.e. whether the metal dopants are well distributed throughout the target or only present as metal clusters at the surface of the target.

The results from the two doping model investigation tests are compared and described in this paper and supported by SEM images that were taken to study the nano structure of the objected ta-C films.

Surface Engineering – The key to a sustainable future & the hydrogen economy

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Sustainability, net zero, and reducing carbon footprints are some of the key areas being addressed by many Governments around the world, but how do we go about achieving these goals? The hydrogen economy has been recognised as a key development in meeting net-zero targets.

Virtually every manufactured product, from aero engines to razor blades, printed circuits to smart phones, utilises a treated surface in some form or other to achieve functional properties that are not present in the bulk material, such as corrosion resistance or wear resistance, or to protect against heat and/or oxidation. Surface engineering provides one of the most important means of engineering product differentiation in terms of quality, performance and life-cycle cost. Its importance to the UK's industrial and financial well-being is beyond question, but what role does surface engineering play in the hydrogen economy of the future.

This presentation, the first of the Quo Vardis Surface Engineering Panel Sessions, will seek to highlight the vital role of surface engineering in the manufacturing supply chain and show how surface engineering really is the key to a sustainable future. It will focus on the use of surface engineering in the hydrogen economy and it will ask if it is possible for HiPIMS to be at the forefront of this journey.

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Tribological performance of doped-DLC coatings produced by HiPIMS in the presence of environmental-friendly additives

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HiPIMS; Ionic liquid; doped DLC; Wear testing.

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One of the major limitations of diamond-like carbon (DLC) coatings applications is related to the low reactivity with oil additives used nowadays. The Ionic liquids (ILs) emerged as a novel class of lubricants that can be used in future lubricated systems due to possesses unique physical properties, including high thermal stability, high thermal conductivity, very low volatility, low melting point, and nonflammability. To improve the lubrication performance of DLC with ILs, it was doped the DLC films with rare earth metals such as Gadolinium (Gd) and Europium (Eu) by High Power Impulse Magnetron Sputtering (HiPIMS). The working hypothesis is that these non-carbide-forming elements can be introduced in the DLC matrix, incorporated as nanoclusters, and enhance the surface adsorption and reactivity of phosphorus-based IL, improving, consequently, the lubricating properties of DLC/ILs sliding contacts, with no effect the mechanical and tribological proprieties of DLC films.

The lubrication of gadolinium-doped diamond-like carbon (Gd-DLC) and europium-diamond-like carbon (Eu-DLC) coatings with trihexyltetradecylphosphonium bis(2-ethylhexyl) phosphate ([P66614][DEHP]) ionic liquid (IL) as 1 wt.% additive in polyalphaolefin (PAO) 8 was studied. The results of the friction tests under boundary lubrication conditions showed that Gd-DLC and Eu-DLC coatings with the IL exhibit a friction reduction, especially with the high atomic concentration of doped metal. Later, the surface results after the long-term wear test indicated that Gd-DLC coatings have less abrasive wear and higher anti-wear properties than Eu-DLC coatings by forming the tribofilm derived from the phosphorus of the IL. In addition, the results of contact angles showed the Gd-DLC coating has high wettability, inferring that the difference between Gd-DLC coatings and Eu-DLC coatings in the friction test with low load can be due to the difference in the wettability. This clearly shows that the novel lubrication system combining the Gd-DLC and Eu-DLC coatings with the IL allows for guiding future research and development.

Evaluating the potential of a CrAlYN/CrN HIPIMS coating to increase the hot forging die life of plasma nitrided H13 steel

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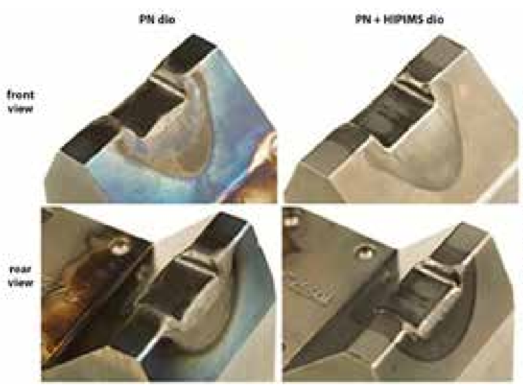
During a hot forging production run, the dies are subjected to cyclical mechanical and thermal stresses. Typically, these will cause the surface condition to deteriorate according to an interplay of damage modes until such point that production must be halted for a die changeover. This invariably has a significant cost impact in terms of time, material, and labour. In industry, the most widely used method of improving the surface durability of hot work tool steel forging dies is nitriding. To the author's knowledge, this is the first time that a HIPIMS coating has been tested for this purpose. The experiments were carried out model dies fabricated from H13 treated by plasma nitriding and duplex treated by plasma nitriding followed by coating using CrAlYN/CrN HIPIMS coating.

The Superlattice structured CrAlYN/CrN with by layer thickness of Δ=3nm was deposited in industrial size Hauzer 1000-4 system enabled with HIPIMS technology at the national HIPIMS Technology Centre UK. Due to the special HIPIMS surface pre-treatment the adhesion of the coating to the plasma untraded surface was excellent of class 1 determined in a Daimler Benz indentation test In high temperature, T= 650C° Pin-on-disc tests using specially prepared counterpart of Inconel 718 the coating showed low coefficient of friction of μ= 0.42 and exceptionally low wear coefficient of K_C = 6.04 x 10^{-13} $m^3N^{-1}m^{-1}$. 260 Inconel 718 billets preheated to 1000 °C were forged onto each in an industrial-scale screw press. The post-forging dies were analysed with a GOM 3d coordinate measuring system, 3d optical profilometry, and SEM with EPMA. Die conditions during the run were inferred from GOM analysis of every fiftieth billet. In terms of local volume loss, the duplex-treated die after 100 forgings was superior to the plasma nitrided die after 260. Moreover, such was the loss from the front edge of the nitrided die, that the rate plateaued after 200 forgings. In contrast, with the HIPIMS coating loss in this region was barely measurable. The plasma nitrided die underwent surface texture, (surface wear) parameter increases of up to 53 times. The highest increase measured for the duplex-treated surface was only by a factor of 3. In a further metric of performance, thermomechanical fatigue cracks were evident on the plasma nitrided die. No such cracks were found on the duplex-treated dies.



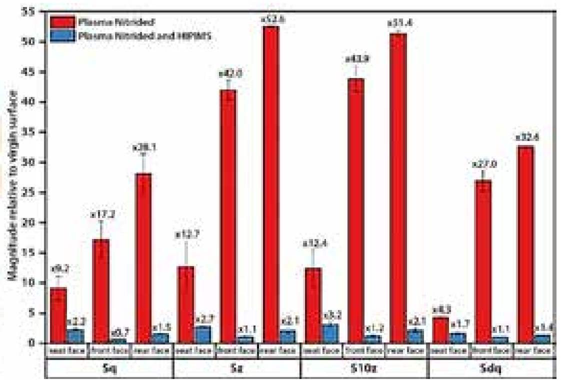


Fig: (left) Photographs showing the post-forging condition of the two dies from front and rear viewpoints.

Fig: (right) Bar chart showing the surface texture evolution of each face of the PN and PN+HIPIMS dies in terms of the relative change of each of four areal roughness parameters.

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Digital Transformation in Thin Film Deposition

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Thin film deposition is an important step within the fabrication of many devices, for example in electronical, optical and tribological systems. In combination with the new development of digital technologies, like data analytics and machine learning algorithms, the ability to control and optimize thin film deposition has been enhanced and with the appropriate infrastructure real-time monitoring and analysis of the processes is feasible. The appropriate infrastructure should handle the integration of data from various devices (cleaning, sputtering, and quality systems) and should have an effective data management system for storing and analyzing large amounts of data.

This presentation shows two aspects within the digital transformation. First, a software framework for automated data acquisition, storing and visualization is presented. It allows to correlate in-situ and ex-situ output data from different sources – including analytical data from coated samples – with process parameters. Second, it is demonstrated how to transform process data obtained experimentally or via physical simulation into a digital twin, which is capable of predicting process characteristics such as the deposition uniformity in real-time speed.

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Structural, mechanical and corrosion resistance of Phosphorus-doped TiAlN thin film

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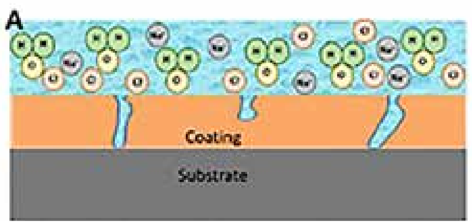
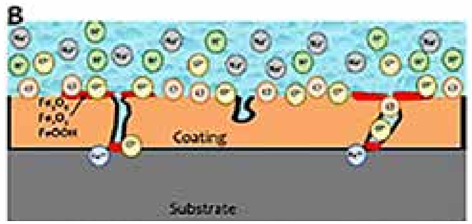
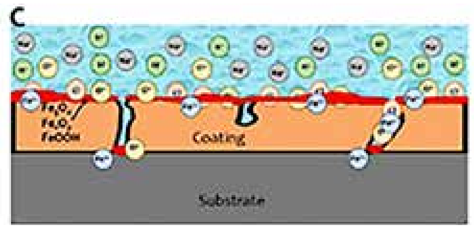
Doped TiAlN thin films are gaining unprecedented attention in recent times due to their functionality and tuneable properties to meet specific demands. The present article focuses on the influence of phosphorus-doped TiAlN thin films deposited using high-power impulse magnetron sputtering. Thin films of different elemental compositions of Ti, Al, and P were sputtered on AISI 5206 steel. The thin film cross-sectional morphology and architecture revealed dense and columnar structures. It was indicated that the (111) diffraction peaks in the XRD pattern shifted to higher angles, while the transverse optics TO)/longitudinal optics (LO) frequency in the optic phonons region of Raman spectra shifted to the right with the modulation wavelength as the Al and P compositions increase. The elementary composition influences the mechanical properties with the maximum hardness of 28 GPa, and adhesion strength of 15 N attained in thin film with the highest Al and P content. The corrosion rate in all the thin films was reduced by at least two orders of magnitude compared with the uncoated samples. The addition of P increases the corrosion resistance of TiAl(P)N thin films.



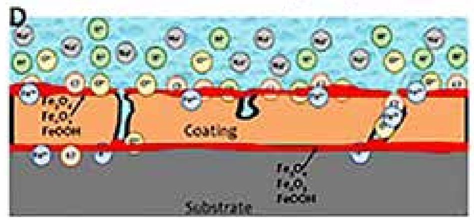
Diffusion of electrolyte through the pores



Pit or localized corrosion taking place at the surface of the substrate through the pores



Propagation of the oxides of iron at the surface of the coating



Galvanic or filiform corrosion taking place at the interface between the coating and substrate

Fig: Schematic diagram of the NSS test corrosion evolution

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Performance and perspectives of roll-to-roll coatings for metallic bipolar plates

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Today's challenges, such as the transition of the energy sector from fossil to renewables, are leading to increasing demands on materials technology and processes with regard to ecological and economic sustainability. Hydrogen-powered vehicles can be one pillar of an independent, ecological and CO2-neutral infrastructure. For the PEM (polymer electrolyte membrane) fuel cell commonly used in the mobility sector, thin stainless steel foil has proven advantageous as a material for bipolar plates (BPP) in terms of cost, weight and potentially recyclability. Currently, the Fraunhofer IWS is working with several other Fraunhofer institutes in a publicly funded project to develop innovative production processes for fuel cells.

In order to enhance the efficiency and lifetime of PEM fuel cells optimized coating systems for the BPP are necessary. The coating must fulfill high requirements for electrical conductivity and corrosion resistance. For this purpose, a roll-to-roll manufacturing concept for bipolar plates is presented with a focus on the PVD (physical vapour deposition) coating development. Coatings from batch and coil processes are compared and new development perspectives for alternative deposition processes are identified.

The comparison in **Fig 1.** shows that both coating variants have a sufficient corrosion resistance as well as a high reduction in contact resistance are achieved compared with uncoated stainless steel. In order to reduce the porosity of the coatings in the arc deposition process and thus achieve a further improvement of the coating properties, developments are being planned in an upcoming project to conduct the deposition using HIPIMS. If the project results are successful, further process development including the transfer to a coal coating system are future research topics.

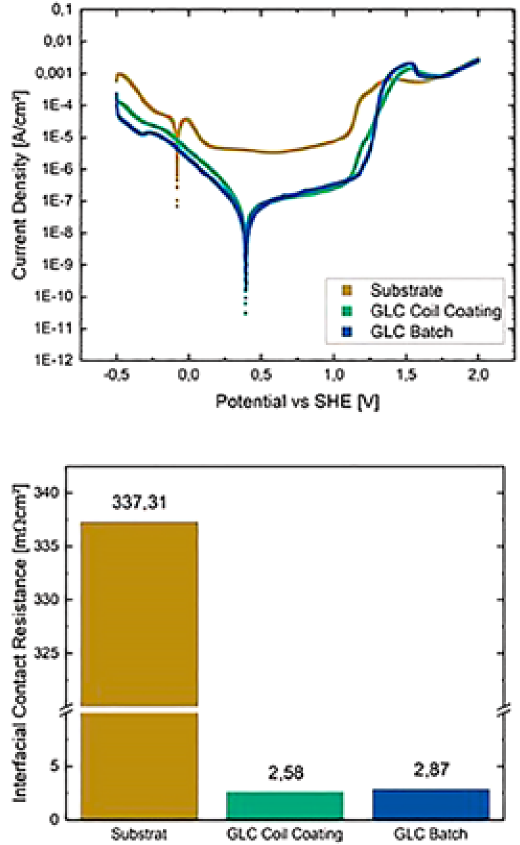

Fig.1: Potentiodynamic measurements (top) and contact resistance measurements (bottom) at 50 N/cm² pressure from GLC coil (Graphite-like carbon) and GLC batch coatings and for comparison the substate (316L)

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The Hydrogen Campus Salzgitter an Innovative Hub for Transformation of the Region

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To tackle the challenges of climate change different initiatives worldwide address similar goals. These measures include the transformation of industry, mobility, the building sector as well as re-establishment and preservation of nature reserves and bio diversity. To combat the challenges hydrogen plays a key role to more sustainable economy.

At the Wasserstoff Campus Salzgitter, the pursued goal is the realization of hydrogen technologies along the entire value chain from creation to utilization, taking especially the economic and environmental dimensions of sustainability into account. Thereby the hydrogen campus servs as a training platform for specialists and executives throughout the region and beyond. Key partners are leading companies (Salzgitter AG, Bosch, Alstom, MAN) as well as research organizations (Fraunhofer IST, TU Braunschweig).

One fundamental goal is the establishment of a model region in the sense of sector coupling throughout the Salzgitter region. This will involve the integration of existing plants for the generation of renewable energies and for water electrolysis. In addition, the local establishment of start-ups in the hydrogen sector is to be promoted.

The talk will give an insight in the developments in the Wasserstoff Campus and the vision behind.

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Ionbond's DOT's technology on titanium bipolar plates for electrolyzers

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The energy transition is accelerating, and the hydrogen economy will be a main player within this change. The hydrogen economy consists of different stages of hydrogen, i.e. generation, storage, transportation, and use. A lot of examples of use already exist, like fuel cells for transportation, hydrogen combustion, fertilizers, house heating, and industrial. The demand is there, but the larger challenge is in generating the hydrogen and getting it to the end users. This paper zooms in on the generation side of hydrogen and more particular the electrolyzer as the enabler for water hydrolysis. The technology is already available for a long time, but we are now in a phase where it needs to be upscaled, made reliable for a long period of time, and produce hydrogen in a cost-effective way. The units are a stack of electrochemical cells, also known as bipolar plates. Today's bipolar plates are typically produced from titanium and protected by electroplated platinum. The key function of the expensive platinum is to protect the titanium from hydrogen embrittlement, while maintaining a low electrical resistance. Ionbond's DOT technology is based on creating small islands, i.e. "DOT's", of either platinum or gold by the thermal spray technology. The DOT's become the contact points for the necessary electrical currents and the hydrogen embrittlement protection film is formed by post oxidation of the not coated titanium surfaces. This technology still uses precious metals, but at much lower and therefor more affordable levels than the incumbent technologies. The electrolyzer performance and lifetime expectation is maintained within this comparison of DOT's and electroplating.

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HIPIMS - The tool for a modern production for coat tools and components

HIPIMS first entered the research stage around the millennium. Starting form the first papers it was intensively explored by academia in different places like e.g. the Sheffield Hallam University, Linköping University or the RWTH Aachen. A lot of advantages of this new technology were seen, but also some challenges in upscaling to large industrial coating machines were discovered: e.g. in the early stages the reliability of power supplies was a hot topic, how to deal with high currents and high voltages in the megawatt range, high heat load on the target, how to do an easy upscale from a lab coater to an industrial size units.

However, the results of improved performance by using HIPIMS technology on cutting tools and components, gained interest of machine builders to overcome the disadvantages and provide solutions. Hauzer was involved right from the beginning of the HIPIMS development with providing solutions and ideas towards industrialization of this technology for manufacturing end users of tools and components.

HIPIMS arrived in today's real industrial scale coating production. Here are the requirement not only providing good coating properties on cutting tools or components, but also production related topics like reliability, easy maintenance, cost per part and flexibility of the coating unit itself plays an important role.

The special properties, that results by using these advanced deposition technology are investigated and demonstrated in industrial automotive, consumer goods and tool applications. The obtained results shown, that the high pulsed technology, HIPIMS, is the right tool for serial production in a modern manufacturing environment. We will also show that this technologies can be easy upscaled on different machine sizes and deliver here reliable industrial processes with a good ratio of coating costs per coated part and performance

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Improvement of Crystallinity and Deposition Rate Using Multi e-HiPIMS

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For two years, we have been studying a new HiPIMS power supply: e-HiPIMS. This e-HiPIMS power supply comprises 3 stages; each stage can be turned on / off and output voltage between 150 and 300 V with 50 V steps. Hence several pulses can be superimposed at different times while the plasma is on. For instance, using two stages to perform a conventional HiPIMS discharge with a pulse duration of 30 µs, the third one can superimpose a pulse of variable voltage and duration. The purpose of this 3-pulse sequence is to take advantage of the hot electrons (so-called 'super-thermal')[1] at the beginning of a pulse to amplify the ionization of specific species. The Langmuir probe measurements confirm the increase in the electron density and the variations in the electron temperature[2]. Two target materials, titanium and chromium, were used to grow thin films with a 3-pulse sequence: conventional HiPIMS (30 µs, 1 kHz) followed by another conventional HiPIMS on which is added a third pulse at the end of the on-time of the previous one. The two conventional HiPIMS pulses of the sequence turn off 25-40 µs after the beginning of the sequence; the first lying from 5 to 15 µs and the second from 25 to 40 µs. SEM analysis revealed the texture of the films of each metal. The last configuration of the 3-pulse sequence leads to the largest grains. XRD measurements showed that the additional voltage (3rd -pulse) significantly improves the crystallinity[3].

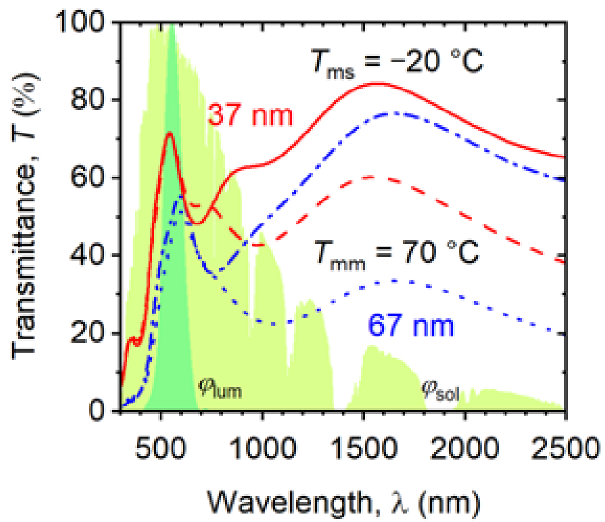
This e-HiPIMS power supply clearly offers promising perspectives unexploited until now, paving the way for fine tailoring the microstructure of the coatings. e-HiPIMS improves the deposition rate with respect to conventional HiPIMS with the same average power. However, the deposition rate remains below the one recorded in DC magnetron sputtering (DCMS), mainly due to the back-attraction of metal ions. By using HiPIMS with very short pulses (less than 5 µs), Antonin et al[4] showed that it is possible to significantly increase the deposition rate (by a factor of two) compared to the classic HiPIMS process. A wide range of 3-pulse sequences of e-HiPIMS confirms the previous findings for Cr and Ti thin film. Notice that the behavior of m-HiPIMS is close to e-HiPIMS, i.e., in both, the burst of hot electrons at the beginning enhances the ionization of residual metal atoms and ions from the preceding pulse[5]. The advantage of e-HiPIMS is the possibility to adjust either the increase of argon ion density or metal ion density, giving more flexibility to the deposition process.

Low-temperature reactive high-power impulse magnetron sputtering of high-performance thermochromic VO_2-based coatings for energy-saving smart windows

Vanadium dioxide (VO_2) exhibits a reversible phase transition from a low-temperature monoclinic VO_2 (M1) semiconducting phase to a high-temperature tetragonal VO_2 (R) metallic phase at a transition temperature of approximately 68 °C for the bulk material. The automatic response to temperature and the abrupt decrease of infrared transmittance without attenuation of luminous transmittance in the metallic state make VO_2-based coatings a promising candidate for thermochromic smart windows reducing the energy consumption of buildings.

To meet the requirements for large-scale implementation on building glass, VO_2-based coatings should satisfy the following strict criteria simultaneously: a deposition temperature close to 300 °C, a transition temperature close to 25 °C, an integral luminous transmittance T_{lum} > 60 %, a modulation of the solar energy transmittance ΔT_{sol} > 10 %, long-term environmental stability, and a more appealing color than yellowish or brownish colors in transmission.

The paper deals with a scalable sputter deposition technique for the preparation of strongly thermochromic YSZ/W-doped VO_2/YSZ coatings on standard soda-lime glass at a relatively low substrate surface temperature (350 °C) and without any substrate bias voltage. The W-doped VO_2 layers were deposited using a controlled reactive deep oscillation magnetron sputtering of a V-W target while the antireflection Y-stabilized ZrO_2 (YSZ) layers were deposited using a controlled reactive standard high-power impulse magnetron sputtering of a Zr-Y target. The fundamental principles of this technique, and the structure and optical properties of the thermochromic coatings are presented. The coatings exhibit a transition temperature of 33-35 °C at T_{lum} = 64.5 % and ΔT_{sol} = 7.8 % for a V0.986W0.014O2 thickness of 37 nm, and T_{lum} = 46.1 % and ΔT_{sol} = 13.2 % for a $V_{0.986}W_{0.014}O_2$ thickness of 67 nm (see the figure). Such a combination of properties, together with the relatively low deposition temperature, has not yet been published by other teams for thermochromic VO_2-based coatings prepared by a scalable technique.



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Non-conventional plasma diagnostics – also for HiPIMS processes?

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Examples for novel "non-conventional" diagnostics, which are applicable in plasma processes, are the determination of the total energy flux from plasma to substrate by calorimetric probes (CP) [1,2] and the measurement of momentum transfer due to sputtered particles or changes of plasma pressure by force probes (FP) [1,3]. These methods will be demonstrated for two examples which are also related to HiPIMS diagnostics.

1.) The combination of high-power impulse magnetron sputtering (HiPIMS) and plasma-based ion implantation (PBII) gives a versatile system which allows successive and simultaneous coating, doping or cleaning of a substrate surface in a single process [4]. Due to the pulsed nature of both processes, the delay between HiPIMS and PBII pulse is a crucial parameter which can be used to effectively tune the process according to the required needs. Since the delay defines at what time during the HiPIMS period the PBII pulse is applied to the substrate, it can also be utilized to obtain time resolved information about the ion density in the substrate's region. To allow direct calorimetric measurement of a high voltage pulsed substrate, a common passive thermal probe (PTP) was modified utilizing a fiber optic temperature sensor. With this probe, the effect of PBII voltage, substrate distance and pressure, on the energy flux and ion current towards the PBII substrate was investigated as a function of the delay between HiPIMS and PBII pulse. A maximum of electrical power and energy flux was observed for delay times significantly longer than the duration of the HiPIMS pulse. The investigation of different PBII pulse durations and PBII voltages confirmed that these parameters affect the absolute values of the energy flux and electrical power but do not significantly affect the transport of the ions as the PBII potential is shielded by a sheath. The results were successfully compared to a basic model and revealed important information regarding substrate position, process pressure and the effect of secondary electrons in this combined system.

2.) A small copper target is sputtered by an ion beam at different angles of incidence. The resulting sputter plumes are characterized directionally resolved using a compact and manoeuvrable force probe [3,5]. The FP measures the momentum flux of particles released due to sputtering or reflection inside the sputter plume. The effect of argon ions at energies from 320 to 1220 eV has been studied. As expected, the directional distributions are sensitive to the angle of incidence. The ion energy has significant influence on the magnitude of forces. The experimental results are compared with simulations (SRIM).

Characterization of a Pulsed Plasma and Macroparticles in an Industrial Scale ta-C Laser-Arc Coating System

Tetrahedral amorphous carbon (ta-C) coatings are commonly used in industry to improve tribological as well as corrosion and wear properties of treated objects. In the early 1990s a new method for the deposition of ta-C was developed, the Laser-Arc [1]. In contrast to other deposition methods, such as IBSD, HiPIMS, other arc setups or combinations, the Laser-Arc technology allows for a strong temporal and spatial control of the arc discharge while providing high deposition rate. This enables up-scaling for industrial applications. A major limiting factor is the generation of macroparticles, see Figure 1, and the resulting defects in the coating. While there are particle filtering systems available, many reduce the deposition rate drastically or require post-treatment polishing which can be expensive, impractical or even impossible [2].

In order to optimize the discharge, reduce the particle generation and improve film growth we measured plasma parameters, neutrals and macroparticles in dependence on arc parameters. The carbon Laser-Arc system produces 100-300 µs, 1-3 kA pulses, which are observed with a custom-tailored diagnostics. Langmuir probes, retarding field analyzers, and optical emission spectroscopy allow for spatial and time resolved measurements of electron and ion energy distribution functions and estimates of neutral densities. Calorimetric probes [3] monitor the energy influx to the substrate which is of special importance when forming tetrahedral bonds. The temporal and spatial macroparticle velocity distributions have been investigated with high-speed cameras.

Fig: A Laser-arc discharge with macroparticles

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Target design for HIPMIS process challenges

Today, many tools and components are coated with different (hard) coatings by utilizing physical vapor deposition methods. The continuous improvement of coating technologies takes place by introducing optimized, combined and more efficient coating deposition methods but also implementation of new material compositions.

These developments require special designs and special materials used for targets.

The leading coating manufacturers work on cost and quality optimization for mass product implementation. The outcome of these efforts is for instance larger coating chambers and shorter process times leading to new target dimensions and shapes as well as the increase in power density applied to the targets. Concurrently, as new coatings and processes become widely accepted, the target manufacturers must support the development and deliver suitable solutions for each requirement.

The targets used for hard coating applications are produced either by powder or by melting metallurgy processes. Targets manufactured by powder metallurgy are characterized by several advantageous properties such as uniform microstructure, high density, as well as homogeneity concerning distribution of chemical elements. The quality of such targets depends on the manufacturing process and for the most part on the quality of the powder ingredients used.

On the one hand, for the development of coatings research is focusing on beneficial effects by alloying with selected elements to control the composition of the coating. The big challenge is to find a suitable technology for production of targets containing all these elements on the one side and to consider the impact of the purity of the targets on the whole production chain and the performance of the final product on the other side.

One the other hand, the development goes also in the direction of optimizing PVD processes, resulting in changing long existing coating processes and think about cost and energy efficiency.

To support the efforts of equipment manufacturers and coating designers, new designs and new technologies must be applied to produce targets in appropriate shape and dimensions. To deliver cost-optimized targets for mass applications the whole process chain, including powder quality and standardization of raw materials, must be considered.

The mentioned efforts, comprising the increase of target utilization, are also strongly related to the increase in power density applied to the targets, which is the case by using HIPIMS technology. Therefore, the development of materials with high heat conductivity and thermal shock resistance are included in the challenges of target suppliers.

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Reactive High Power Impulse Magnetron Sputtering (HIPIMS) of titanium oxide: Transition from metallic to poisoned regimes analyzed by optical emission spectroscopy.

The hysteresis behavior of target poisoning during reactive High Power Impulse Magnetron Sputtering (HIPIMS) of Titanium in Ar/O^2 atmosphere has been investigated using a dual cathode arrangement (length per cathode approx. 500 mm) in an industrial in-line coating system. The deposition process was studied at constant pulse voltage (up to 1.1 kV) and constant average power (P_{av}) by adjusting the repetition frequency. The current and voltage of the power supply as well as optical emission spectra in the range of 200 to 1100 nm were monitored and analyzed.

As reported in literature, also for large cathodes it was found that the target peak voltage and pulsing parameters affect the overall shape of the hysteresis loop. For all experimental conditions, adding oxygen to the sputtering process led to a change in emissions peaks of Ar, O and Ti and Ti^+. This depends on the Ar/O_2 - ratio and the condition of the target surface. Pure titanium shows a saturation in peak current in contrast to poisoned target which show a higher peak current and a triangle form of the current over pulse time. The data from peak current and emissions lines in combination were used for controlling the process to deposit TiO_2 thin films.

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Oxidation behaviour of HIPIMS and DC magnetron sputtered (Hf1-xAlx)By thin films

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Hafnium diboride (HfB2) is an ultra-high temperature ceramic and it is showing the best oxidation resistance among transition metal diborides (TMB2). Adding Al to TMB2 can further improve the oxidation resistance. By using HIPIMS and DCMS to deposit (Hf1-xAlx)By thin films, this study explores systematically the effect of the sputtering technique and the influence of the boron stoichiometry y = B/(Hf+Al) on the oxidation resistance. Therefore, a three-target setup including two compound HfB2 and AlB2 targets and one elemental Al target was used. While the compound targets were constantly run in DC mode, the Al target was operated in HIPIMS for selected depositions. In this way, the influence of the HIPIMS plasma in comparison to the DCMS plasma on the phase formation and the solubility of Al in HfB2 was studied systematically. Deposition conditions were varied to obtain understoichiometric (y < 2), stoichiometric (y = 2), and overstoichiometric (y > 2) (Hf1-xAlx)By thin films. Afterwards, the thin films were oxidized at 700 °C in the air for up to 8 h and subsequently analyzed by different characterization methods. X-ray diffraction (XRD) was used to study the formation of new phases and oxides upon oxidation in air, while scanning transmission electron microscopy (STEM) was performed to examine the microstructure and the oxide layer thickness. First results indicate a significantly reduced oxide layer thickness of overstoichiometric (Hf1-xAlx)By thin films compared to (Ti1-xAlx)By thin films. Elastic recoil detection analysis (ERDA) and X-ray photoelectron spectroscopy (XPS) were utilized to determine the chemical composition and the chemical state of the oxide layer formed upon oxidation. In summary, this oxidation study of (Hf1-xAlx)By thin films contributes to understanding the possibilities of HIPIMS to tune material properties.

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Al-rich (Al,Ti)N at the cutting edge: Insights into the incipient nucleation of the B4 phase

More than 35 years after their first description [1], NaCl-type B1 titanium aluminum nitride films remain the most widely adopted PVD material for protective tool coatings. A successive increase in Al-content brought about improvements in oxidation resistance, hardness and overall wear behavior. However, any extrapolation beyond state of the art Al-contents of $c_{Al}/(c_{Ti}+c_{Al}) \approx$ 60-67% is complicated by a progressive decrease in the free enthalpy of the wurtzite-type B4 phase, whose formation is commonly associated with a deterioration of cutting performance [2].

In the present study, the issue of higher Al-content (Al,Ti)N is revisited via a correlative analysis of HiPIMS coatings, that is based on spatially resolved nanoindentation. It is shown that (cutting) edges as geometrical features of the coated substrate play an important role in preserving a cubic growth. This is commonly reflected in a characteristic spatial distribution of phase composition: Emanating from the edge line, a purely cubic region of variable spatial expansion may be obtained. However, with increasing edge line distance the drop in electrical field enhancement ultimately leads to the formation of B4 phases correlating with an overall structural refinement. Based on HR-(S) TEM this progressive refinement is attributed to a competitive surface nucleation of B1 and B4 crystallites.

Rather than detrimental to the cutting performance, this allotropic nucleation may be conceived as a means to tailor the properties of (Al,Ti)N coatings. Applications of high Al-content (Al,Ti)N coatings featuring the above characteristics are briefly introduced.

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Tuning Residual Stress Depth Profile for Enhanced Tribological Properties of TiAlN Coatings Prepared by HiPIMS Using a Cylindrical Magnetron

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In the present work, we systematically study the effect of the coating growth conditions on the development of residual stress (RS), especially on the RS depth profile between the coating surface and the coating-substrate interface. This investigation is meant to develop approaches to mitigate RS in general, and to take it one step further: to tailor the stress profile to better oppose stress distribution imposed by different levels of loading during tribological conditions applied in real-life applications. TiAlN on WC-Co substrates was used as a model system.

The coatings were prepared using $Ti_{0.5}Al_{0.5}N$ compound targets in two setups, one equipped with a planar magnetron (PM), and another one with a 500 mm long cylindrical magnetron (CM). *In situ*, real-time monitoring by optical emission spectroscopy (OES) was applied for the study of the process and film growth conditions.

Structural characterization and RS measurements were performed using multi-hkl grazing incidence X-ray diffraction (MGIXRD) in a standard laboratory XRD system as well as in the Canadian Light Source X-ray radiation synchrotron facility. Tribo-mechanical properties were measured by depth-sensing indentation, pin-on-disc and ball-on-flat tribometers, and micro scratch testing, while further analyses were performed by optical profilometry, SEM and EDS.

In the first part, we studied a **single-layer system** while controlling the substrate bias (V_B). Hardness increased from 20 GPa (no bias) to 30 GPa for V_B = -60 V without any heating. This correlates with the increase in compressive RS from -0.9 GPa to -5.5 GPa and the corresponding decrease in the grain size (from 16 nm to 9 nm). The RS depth profiles clearly show a steep gradient in RS increasing from the interface to the surface. Substrate heating enhanced the mechanical properties, accompanied by a lower compressive RS and its gradient. Combined substrate temperature (350oC) and biasing gave rise to even lower stress (-2.3 GPa).

In the second part, two film architectures were investigated, namely a single TiAlN **layer with a graded interface** due to V_B variation, and a **14-layer architecture** when individual layers were deposited at different V_B between -60 V and -100 V. RS values varied from -2.0 to -7.6 GPa that strongly correlated with the tribological performance. Specifically, high stress close to the surface reduced the abrasive wear by hindering crack initiation and propagation and contributes to low wear rates for short sliding distances. In contrast, lower stress at the interface led to high wear rates for longer sliding distances. The TiAlN coatings outperform the substrate at high temperatures (700 °C) due to its severe oxidation. Wear rate of the coating increased with a longer sliding distance, possibly due to a reduction of the RS at higher depths.

The results clearly show that tuning the residual stress depth profile can effectively be used to tailor the mechanical and tribological properties of the TiAlN (and other) protective coatings for enhanced performance at specific application-related loading conditions.

High volume magnetron sputtering: time and cost effective technology to manufacture critical components in PEMWE

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PEM electrolysis is a technology that is gaining momentum among the different existing electrolysis technologies for the storage of renewable energy in the form of H2 due to its ability to respond efficiently and quickly to fluctuations associated with the generation of electricity from renewable energy sources (wind and photovoltaic). This advantage is combined with their capability to operate at high current densities, high efficiencies, excellent partial load behaviour, together with simplicity of plant balance and design compactness.

Currently, the main challenge related to the technology is to reduce manufacturing costs without penalizing efficiency, reliability and durability. For that purpose, the substitution or reduction of expensive materials (such as Pt, Ti, Ir) is being investigated as well as industrial techniques able to manufacture these components through time and cost-effective processes.

This work presents high volume magnetron sputtering technology as ideal option for dry, automated and mass production of:

- A low Pt loading electrode for hydrogen evolution reaction (HER). Thermogravimetric analysis (TGA) shows a reduction of 56% in Pt loading when compared with a commercial Pt black electrode with a Pt loading of 0,3 mg/cm^2. Electrochemical characterization has been also carried out through linear sweep voltammetry (LSV) and cyclic voltammetry (CV) to determine the overpotential at 10 mA/cm2, stability and ECSA (electrochemical surface area) of the new electrodes.

- PGM-free coating alternatives for stainless steel BPPs deposited using HIPIMS technology. Their electrochemical performance (potentiostatic and potentiodynamic measurements) has been investigated in simulated PEMWE anode conditions as well as during in-situ testing in a $25cm^2$ single cell under real PEMWE conditions.

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Carbon hybridization states quantification by EELS and NEXAFS in hard DLC films deposited by HiPIMS-DOMS

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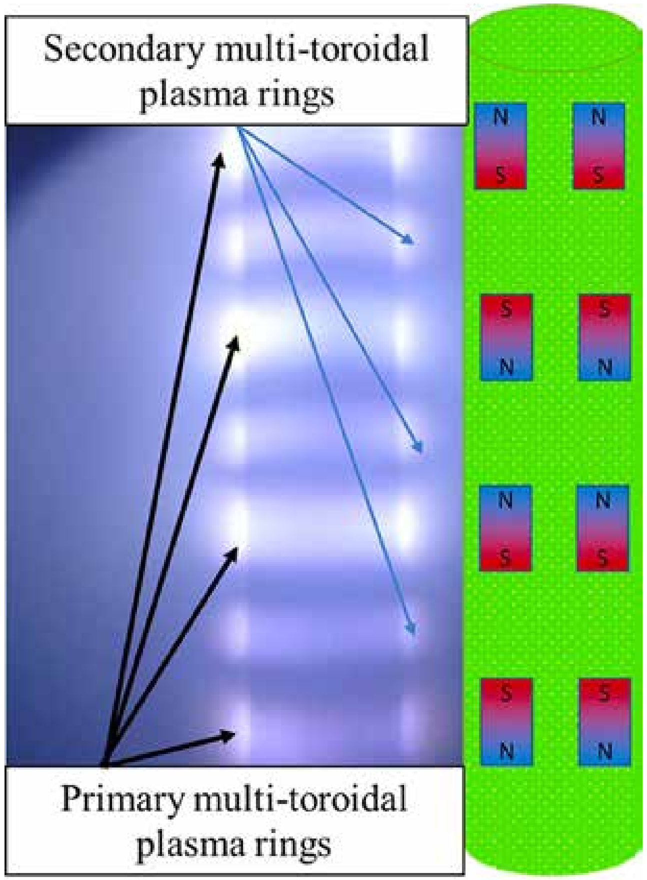
Diamond-like carbon (DLC) thin films are widely used in many industrial applications, including cutting tools, mechanical seals, and wind turbines, mostly to increase energy efficiency by reducing friction and wear. The most significant driving factor for the DLC market is the rising demand of DLC coatings for automotive engine parts. However, DLC coatings application in combustion engines is hindered by their limited thermal stability. Upon heating, the sp3 -type bonds of DLC revert to sp2, causing the DLC films to lose their unique combination of high hardness and low friction coefficient. Thus, quantifying the percentage of sp2 or sp3 bonds in a DLC films is of paramount importance to design DLC films and tune their deposition processes. Many advanced analytical techniques, such as X-ray Photoelectron Spectroscopy (XPS) and Raman Spectroscopy, have been employed to evaluate the sp3 content in DLC films. However, these techniques rely on an indirect information on the sp3 bonds and thus are qualitative rather than quantitative. In this work, DLC films were deposited by Deep Oscillation Magnetron Sputtering (DOMS), a variant of High-Power Impulse Magnetron Sputtering (HiPIMS), using both pure Ar and mixed Ar + Ne plasmas. The local structure of the DLC films were first characterized by Raman spectroscopy and their mechanical and tribological properties were evaluated by nanoindentation and pin-on-disk tests in the as-deposited state and after annealing in protective atmosphere up to 700 ºC. The amount of sp3 sites in DLC films has been measured by Electron Loss Spectroscopy (EELS) and Near Edge X-ray Absorption Spectroscopy (NEXAFS) which provide detailed quantitative information about the carbon hybridization states. The DLC film were annealed ex-situ in vacuum up to 700 °C and in-situ during the NEXAFS measurements up to 900 °C. The sp3 /sp2 ratio in the films was evaluated both as a function of the film thickness and annealing temperature. Additionally, the structural order of the films has been assessed by High Resolution Transmission Electron Microscopy (HRTEM). All the deposited DLC films have a quasi-amorphous structure. Adding Ne to the plasma increases both the hardness and the thermomechanical stability of the DLC films. The sp3 content in the DLC film deposited in mixed Ar + Ne plasma is close to 40 % along the whole film thickness. The sp3 content in the film deposited in pure Ar varies significantly with the film thickness. It slightly increases from the bottom to approximately half of the film thickness, from 27 to 30%. However, at higher thicknesses, the sp3 content decreases gradually with increasing film thickness down to close to 18 % near the top of the film. The sp3 content in the DLC films shows an exponential decrease down to close 20 % with increasing annealing temperature up to 700 and 800 ºC for the DLC films deposited in pure Ar and with Ne addition to the plasma, respectively. For both types of films, the sp3 content sharply decreases towards zero at higher annealing temperatures due to complete graphitization of the films.

Comparative study of DC and HIPIMS discharge characteristics of Cr cylindrical magnetron

Cylindrical magnetrons are sources of non-equilibrium plasma generated on the outer or inner surface of cylindrical cathodes. Multi-toroidal plasma geometry generated on the outer surface of cylindrical cathodes allows to deposit coatings on surfaces inaccessible to standard planar magnetrons like the interiors of pipes or barrels. Direct current magnetron discharge sources are often characterized by current-voltage characteristics of the form $I \propto V^q$. It has been suggested that the exponent q provides an index to the effectiveness of the magnetic electron confinement in a magnetron discharge[1]. An application of a magnetron in a confined space can significantly influence the magnetic field and thus the electron confinement. Therefore, we have examined the I-V characteristics of DC, pulsed DC and HIPIMS discharge of Cr cylindrical magnetron with multi-toroidal plasma geometry in Ar atmosphere in open and confined space realized by a A570 Gr. 36 steel pipe. The q parameter of the exponential function Id (Ud) describing current-voltage waveforms was determined. The study confirms that the type of discharge and magnetron surroundings significantly affects the value of q.



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Novel approaches for the PVD synthesis of advanced aluminide thin films: The example of Ruthenium-Aluminide

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Considering high temperature applications, aluminide intermetallics achieved increasing importance over the last decades. They are well known for their mechanical properties, such as high melting point, strength and good oxidation resistance. In Ni-superalloys, for example, aluminide precipitations are widely used as toughening phase, increasing the high temperature strength and durability of the construction material. Although they are commonly used as an additive in composite materials, their usage as a bulk material is hindered by their poor manufacturing due to its brittle behaviour at room temperature.

A relatively new candidate material of B2 structured aluminides is the RuAl intermetallic phase. Compared to other candidates of its class, such as NiAl or TiAl, RuAl exhibits a ductile-brittle-transition below room temperature, which may considerably expand the range of its potential applications.

Thin film synthesis can enable the exploitation of their full potential for example as a protective coating in aircraft and aerospace applications. To elucidate this potential, RuAl thin films were synthesized by magnetron sputtering, utilizing different approaches. For the deposition of single layer thin films, a powder manufactured sputtering target with a composition of 50 at. % Ru and 50 at. % Al was used to directly sputter the desired phase. A more complex route is the usage of nanoscale multilayer thin films, showing the desired phase formation by thermal post-treatment. Thin film deposition was done for a variation of the process parameters such as the mode of the power supply, gas pressure and bilayer thickness (for multilayer thin films) to investigate their impact on the thin film constitution and microstructure. Major structural thin film characterization was done by X-ray diffraction and transmission electron microscopy methods. These data are subsequently used to discuss the mechanical properties of the thin films, determined by microindentation.

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PVD coating in the production environment Industry 4.0

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iwis mobility systems GmbH & Co. KG is part of the iwis Group with headquarters in Munich. The German family-owned company with 50 locations worldwide and 3,200 employees acts as an international technology leader and manufacturer of precision chain systems, mechanical drive technology as well as connection and contact technology. With this product portfolio, the company achieves perfect precision and continues to lead the automotive sector towards the future in the areas of e-mobility and autonomous driving.

PVD series coating of miniature components in mass production: Another lesser-known mainstay of the Munich-based company is the product-specific surface finishing technology "TRITAN" developed almost 12 years ago in the context of the development of diesel engines. Modifications to the framework parameters, including the extension of maintenance intervals, changed the characteristics of the soot particles and the acid ingress in the oil. This change led to higher demands in terms of wear resistance and thus to the necessity of implementing an improved coating of the pins in the timing chains.

The core of the technology is PVD coating with the "Triboliner" system from Hauzer.

The entire process chain of the coating process is fully automated. The Industry 4.0 process ensures unambiguous traceability down to the smallest transport unit - the workpiece carrier in the coating process - and is of course specially designed for small components and high volumes - up to 500,000 pieces (pins) per day & plant. Conversely, this means coated bulk surfaces with the highest quality standards in the automotive environment and equally high precision for the customer.

TRITAN®

The PVD coating process from iwis enables the seamless coating of rotationally symmetrical components without a fixture. In doing so, it offers new cost standards for small components by significantly increasing production volumes.

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Constitution and properties of TiC1-x:H/a-C:H nanocomposite thin films prepared by HiPIMS processes at low and elevated temperature

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HiPIMS, Carbon-based nanocomposites, ERD, XRD, Raman spectroscopy, Vickers hardness

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Reactive HiPIMS processes can be used to selectively prepare functional coatings in the Ti-C-H system with different constitutions and microstructures. A titanium target is used and, with the argon working gas flow kept constant, the CH_4 reactive gas flow is systematically varied. The HiPIMS synthesis is carried at substrate temperatures of 120°C and 400°C in order to investigate the effect of deposition temperature on phase formation. With increasing CH_4 reactive gas flow, the following structures are realized at both low and elevated deposition temperatures: pure Ti coatings, substoichiometric single-phase nanocrystalline fcc TiC_{1-x}:H thin films, nearly stoichiometric single-phase nanocrystalline fcc TiC:C,H coatings with C and H at grain boundaries, and nanocomposite coatings consisting of nanocrystalline fcc TiC:H,C and a-C:H. At higher deposition temperature, lower CH_4 reactive gas flux is required to obtain similar bonding structures, the crystallites are larger and hydrogen incorporation into the crystallites and accumulation at the grain boundaries is lower. All layers are characterized by ERD, XRD and Raman spectroscopy with respect to their structure and the Vickers hardness as a mechanical property is investigated in detail.

Real-time monitoring of early stages of HiPIMS film formation on polymers with GISAXS

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High-power impulse magnetron sputtering (HiPIMS) is a well-established coating process that provides improved and specialised coating properties compared to regular DC magnetron sputtering (DCMS) (1, 2). The key features responsible for these advantages are the high degree of ionization and increased kinetic energy of target material. Our aim is to investigate the relation between the distinct features of the HiPIMS process and the film formation during the early stages of material deposition. The main diagnostic for that purpose is grazing incidence small-angle X-ray scattering (GISAXS) performed at P03, PETRA III, DESY. This technique allows the in-situ monitoring of even sub-monolayer nanostructures with a high temporal resolution and good statistics due to the large probed surface area. The size, shape and density of clusters on the substrate surface can be followed during deposition until a closed layer is formed. Additional acquisition of in-situ UV-Vis data allows the correlation of optical properties, e.g., absorption due to surface plasmon resonance or band gap. Comparable experiments with metals on different polymers with DCMS were performed in the past (3, 4) and serve as a well-established basis for modelling and comparison of the data.In-situ measurements were conducted on model polymers like spin coated polystyrene (PS) and Poly (methyl methacrylate) (PMMA) as well as polymers deposited by initiated chemical vapor deposition (iCVD), e.g., tetravinyl-tetramethylcyclotetrasiloxane (V4D4) and ethylene glycol dimethylacrylate (EGDMA) as hydrophobic and hydrophilic specimens. The distinct functional groups of these polymers influence the growth in different ways. Sputter materials used are Au, Ag and TiO2 (reactively sputtered). The results show a pronounced influence of the sputter technique used on the early nucleation stage. For HiPIMS, the number of clusters is increased, i.e., the cluster size is reduced, compared to DCMS. Increasing the peak current further amplifies this tendency. This observation is probably linked to the creation of defects on the polymer surface due to ion bombardment as additional starting points for nucleation. These results are verified with UV-Vis and SEM measurements. Additional adhesion tests show their influence of deposition parameters on a macroscopic scale.

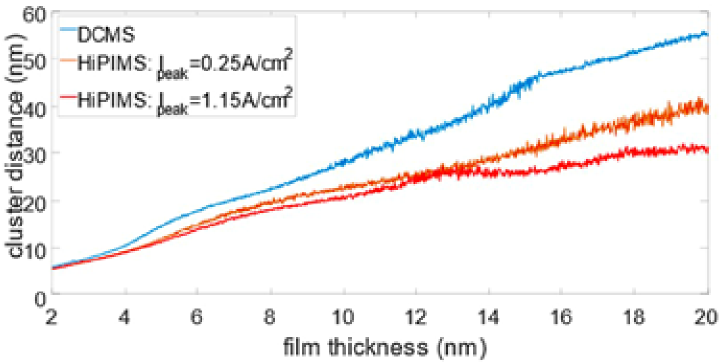

Fig: Cluster distance vs. film thickness of Ag deposited by DCMS and two different HiPIMS parameters on V4D4.

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Effect of metal oxide dielectric layers deposition by High Power Impulse Magnetron Sputtering on Resistive Random Access Memory performance

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In this work reactive magnetron sputtering using HIPIMS discharge was used to deposit metal oxide thin films. It was selected and tested four metals to prepare its oxides, as follows: aluminum oxide (AlOx), titanium oxide (TiOx), zirconium oxide (ZrOx) and hafnium oxide (HfOx). Those oxides play very important functions in various structures for novel electronic and photonic devices. In this case the metal oxides films are used for dielectric layers in MIM (Metal Insulator Metal) structures. Such structures are the basis of resistive random access memory (RRAM) application.

There are compared structures fabricated by the standard pulsed DC and HIPIMS technology, based on the thickness, refractive indices, transmittance and reflectance in the UV-VIS range. The resistive switching properties of the MIM structures with the employed oxide materials depend on the presence of oxygen vacancies in the layer bulk. In order to monitor the stoichiometry of the oxide layers, MIS (Metal-Insulator-Semiconductor) structures are fabricated. The analysis of the obtained electrical characteristics was performed. In the last part of this work, selected processes were used to fabricate MIM devices.

The results of the electrical characterization of the fabricated test structures will be described indicating concluding remarks on the feasibility of applying the studied structures in RRAM devices.

Advanced Process Control for Metallic and Reactive HIPIMS Applications for Production Lines

The majority of vacuum coatings are compound materials. Deposition from compound targets with low deposition rate and deposition from metallic targets with reactive gas at lower costs are in competition for industrial applications. For the latter, active process control is required for cost-effective production, but is still avoided in industrial plasma processes despite major progress in hardware, software and data evaluation of commonly known control techniques. Optical emission spectroscopy is well known for decades to monitor and control plasma processes but it is still "unwanted" in production lines. However, plasma processes are becoming more complex, e.g. like HIPIMS and highly ionized plasmas, and production processes are facing more demanding requirements towards production quality, production stability, production yield, and cost efficiency. At the same time, control techniques such as spectroscopic plasma monitoring have been improved considerably in the last years with respect to reliability, maintenance, and operation and are ready to be used in production lines meeting the challenges of recent production processes. The latest achievements of the plasma monitoring technique are presented for metallic and reactive HIPIMS processes demonstrating the simultaneous control of ionization degree and reactive gas flow not only for small cathodes but also for large area coatings with sectional balancing of the reactive gas flow. For all non-reactive highly ionized applications the degree of ionization can be pinned by the peak current and controlled by spectroscopic plasma monitoring. The corresponding algorithms are realized in a single control system with industrial interfaces for full integration in the overall production control system. Examples of HIPIMS processes for TiO2, SiN, and DLC applications addressing long-term stability and target erosion are presented.

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Prediction of OES data based on HPPMS process parameters by ANN

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High Power Pulse Magnetron Sputtering (HPPMS) processes are known for their high peak powers and voltages, leading to a high concentration of ionized metal ions in the coating plasma. Furthermore, the plasma properties have a significant influence on the coating properties, like the chemical composition. Thus, knowledge of the plasma properties during the coating process is crucial. To understand the relationship between parameters of the coating unit and intensity of the plasma species, optical emission spectroscopy (OES) can be used. However, current models for predicting plasma parameters based on coating unit data are complex and cannot fully account for all correlations. Artificial neural networks (ANN) can be used to identify these correlations and predict OES data and plasma properties respectively using data from the coating machine. In this study, various coating processes using elements such as Al, Cr, Ti, N, and O were investigated, using cathode current and voltage, substrate bias, chamber pressure, gas flow, and target composition as input parameters for the ANN. The output data was time-resolved OES data for metal and gas species. To determine the most effective training algorithm for these predictions, multiple algorithms were tested. Most promising results were obtained for the Levenberg-Marquardt and the One-Step-Secant algorithm. Good prediction accuracy was obtained for TiAlN coating processes and for the intensity ratios of gas species in CrAlON coatings. Processes based on high performance coating plasmas can be developed in a shorter time by using ANN, since the amount of needed experiments can be reduced. By combining OES and ANN the development costs for coating processes can be reduced.

Thick and smooth nanostructured Cr-based coatings by HiPIMS

The atomic shadowing effect is one of the main factors that undermine the performance and limits the maximum achievable thickness of films deposited by magnetron sputtering processes. The effect, which always occurs in sputtering during film growth, originates from the preferential deposition of obliquely incident atoms on higher surface points of any substrate. The atomic shadowing effect drives the formation of an open columnar anisotropic microstructure, with columns interspersed with voids or underdense regions, and increases the surface roughness as the film´s thickness increases. Intense energetic ion bombardment during film growth can suppress the columnar microstructure development by counteracting the atomic shadowing effect. However, bombarding the film's surface with highly energetic particles comes with a heavy cost: formation of a high density of defects, which disrupts the crystalline structure of the coatings, and generation of high compressive stresses. These cause degradation of the films crystalline quality and properties, resulting eventually in premature film failure during service. Counteracting the atomic shadowing effect by using ionised sputtered species is proposed to avoid the bombarding problems. Ionisation of the sputtered species at low peak power can be used to control the impinging angle distribution of the species arriving at the substrate during film growth without increase of their energy. The atomic shadowing effect is this way reduced, or even completely supressed, by reducing the high angle component of the impinging species angle distribution. Thick chromium films were prepared by deep oscillation magnetron sputtering (DOMS), a variant of high power impulse magnetron sputtering (HiPIMS), with different levels of ionization to test and study the influence on the film growth conditions and respective coating properties, like structure and surface morphology. An electrostatic flat probe mounted at the substrate location was used to characterize the saturated current density of positive charges bombarding the substrate during film growth, evaluating the flux of positive ions bombarding the growing film. Film hardness decreases with increase of thickness, however, Young's Modulus values remain close to Cr bulk value. The films have [110] preferential orientation depending on the bombarding conditions.

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Thin film materials design & some thoughts on complexity and sustainability

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Designing the next generation of thermally stable thin films without utilizing trial and error based methodologies requires truly predictive computational approaches. Important design criteria for protective thin film materials are the mechanical behavior as well as thermal and chemical stability.

In this talk an effort is made to describe the good, the bad and the ugly of our predictive capabilities today. Examples of predictions that have been validated experimentally and experimental data that cannot be described theoretically will be presented. Implications for future design efforts will be discussed also in the context of (chemical and structural) complexity as well as sustainability.

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Solid Solution and Ordered Quaternary MAX Phase Thin Films: Phase Formation and Reaction Mechanisms in Magnetron Sputtered Nanostructured Multilayers

Mn+1AXn (MAX) phases are nanolaminated compounds that possess remarkable and unusual properties combining attributes of both metals and ceramics. Multi-element alloying strategy offers further degrees of compositional and structural control to increase their chemical versatility and expand their physical and chemical properties. Double transition metal alloying to synthesize quaternary phases has resulted in the discovery of many new solid solutions and ordered phases (including out-of-plane ordering and in-plane ordering in M-layers). Intensive efforts are currently ongoing to explore new approaches for synthesizing high-quality ternary MAX phase thin films, but the synthesis of quaternary MAX phase thin film materials has been rarely explored yet. In this study, we focus on synthesis of quaternary (M^1M^2)n+1AXn thin films in three different systems, i.e. Cr-M^2-Al-C with M^2: V, Ti, Zr, via thermal annealing of nanoscale multilayered thin film precursors. These material systems cover the three types of chemical ordering on M-layers: random solid solution (CrxV1-x)2AlC (0 < x < 1), out-of-plane ordered $(Cr_{2/3}Ti_{1/3})_3AlC_2$, and in-plane ordered $(Cr_{2/3}Zr_{1/3})$2AlC. The multilayered thin film precursors with pre-defined nanostructured architectures and varied chemical compositions comprising large fractions of Cr - M^2 binary systems were deposited by combinatorial magnetron sputtering using segmented transition metal targets, and pure graphite and Al targets. Their temperature-dependent phase formation during subsequently annealing in argon has been investigated by in-situ high-temperature X-ray diffraction (HT-XTD) and ex-situ XRD. Thorough microstructural and compositional analyses at the nanoscale, combing high-resolution scanning transmission electron microscopy (HR-STEM) and atom probe tomography (APT), were performed to gain a fundamental understanding on the thermally induced phase evolution and microstructural formation. Solid solution (CrxV1-x)2AlC and out-of-plane ordered $(Cr_{2/3}Ti_{1/3})_3$AlC2 were successfully obtained after annealing at specific temperatures, while annealing of the multilayered precursors in Cr-Zr-C-Al system mainly resulted in the formation of ternary MAX phases and binary compounds. The reaction paths and reaction mechanisms towards the complex quaternary MAX phase structure formation in the three different systems will be discussed, which provides guidelines on designing suitable nanostructured multilayer precursors for synthesizing high-quality quaternary MAX phase thin films.

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Effect of current-voltage characteristic on plasma ionization in HIPIMS discharge of graphite target in argon atmosphere

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Diamond-like coatings (DLC) have attracted much attention as a coating for a wide range of applications due to their properties such as excellent frictional behavior, high hardness, elastic modulus or biocompatibility. These properties are related to the microstructure, i.e., sp^2 and sp^3 bonding ratio in the carbon network. In the conventional sputtering methods, plasma density is relatively low so that the ionization effect is small, resulting in the low sp^3 fraction in DLC films. High Power Impulse Magnetron Sputtering (HIPIMS) has attracted much attention in DLC coating deposition because of high ionization effect of sputtered material. High carbon ion density in HIPIMS discharge plasma leads to tetragonal bonds of carbon atoms which results in higher hardness and better wear resistance of the coatings. However, a common issue in C sputtering is applying long pulses which lead to the occurrence of arcs and decrease in coating quality. This effect is even more prominent in high current discharges such as HIPIMS discharge. A way to mitigate arc occurrence is to use high frequency pulses with short pulse time.

This work focuses on determining the effect of the duty cycle, pulse time, frequency and current on the carbon ionization. Using a graphite target, OES spectrums of HIPIMS discharge in argon atmosphere for various power supply parameters was observed. Initially, peak current density was increased from 0,5 to 2A/cm². Following, pulse time and frequency were varied between 5 – 200 µs and 300 – 10’000 Hz respectively. Finally, HIPIMS OES spectra were compared to pulsed DC discharge spectrum.

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Novel Sputter Cathode Design to Obtain Very High Ionized Flux Fraction of Deposited Material in Industrial Conditions

An extremely flexible coating unit Platit Pi411 PLUS enables a wide spectrum of PVD and PECVD coatings thanks to its several different cathode configurations. Besides cathodic arc evaporation, this unit also offers magnetron sputtering by a central cylindrical rotating sputter cathode situated in the middle of a chamber. There are two different types of sputter cathode in Platit's portfolio – well-established Platit SCIL cathode and novel Platit F-Type cathode. These cathodes will be described in more detail and compared in terms of their specific properties.

The Platit SCIL cathode has a conventional cylindrical arrangement of the magnetic field and is designed for standard DC magnetron sputtering. For a titanium target sputtered in an argon atmosphere at 0.5 Pa pressure and power of 25 kW, the discharge current is 52 A and the ionized metal flux fraction of titanium on a substrate goes up to 20 %. Platit's second sputter cathode F-Type is the industry's answer to HIPIMS, where it is not necessary to acquire a new power supply. Instead, the Platit F-Type operates on a new principle where the plasma is concentrated to a significantly smaller volume compared to Platit SCIL by magnets which perform an oscillating longitudinal movement inside the cathode to spread the plasma over the entire length of the cathode and at the same time the target is sputtered symmetrically in 360°. This different construction dramatically changes the shape of a racetrack while still enabling it to deliver a high DC power of 25 kW. F-Type reaches discharge current up to 65 A. The ionized metal flux fraction at a power of 23 kW is 35 % which is comparable with HIPIMS operation.

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Properties of hard C coating prepared by different HIPIMS processes.

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A description of different PVD processes for the preparation of hard carbon coating is presented. The focus is dedicated to HIPIMS, including the new hybrid PVD deposition system based on the combination of HiPIMS pulsed glow discharge and pulsed cathodic arc discharge called HiC-ARC.

The presence of cathodic arc discharge in the HiPIMS is a typical situation when the glow discharge randomly transits to an arc discharge during the active part of the pulse cycle. In these typical cases, the transition appears randomly and cannot be controlled. The arc can live for a long time and emit macroparticles that can deteriorate the properties of the coating. The new hybrid HiC-ARC discharge works with modified pulsed high power supplies, which allow the initiation of pulsed arc discharge during HiPIMS pulse at the defined time. The initiated arc during the active part of the HiPIMS pulse is quenched at the end of this active part of the pulse when the magnetron cathode is disconnected from the negative voltage of the power supply. This results in a controlled number of macroparticles in the growing films. Several modifications with the HiPIMS sequence of active pulses with arc were tested as well. Three regimes in the measured waveforms were found – HiPIMS regime, enhanced HiPIMS regime, where high currents were measured, but the arc was not ignited, and arc regime.

Plasma diagnostics was done in the HiPIMS-based system by an energetically resolved ion mass spectroscopy and by a RF ion flux density monitor, as both methods worked with the time resolution. Ion energy distribution functions of C^+, C^{++} Ar^+, Ar^{++} were observed at the position of the substrate for different deposition conditions.

Carbon ta-C films were deposited at different working gas pressures and pulse configurations. Deposited films were analysed by DUV and VIS Raman microspectrometer and microhardness measurements and the ratio of sp^3/sp^2 bonds was estimated. The surface morphology was obtained by confocal microscopy, and the presence of microdroplets was analysed for particular conditions. Achieved ta-C film parameters for this deposition source were compared with other deposition methods, especially DC arc plasma. The carbon target was weighted prior to and after the deposition, and the energetic balance of the process was calculated.

The deposition rate of the dielectric films prepared using reactive high-power impulse magnetron sputtering can be very high

Two main problems in a standard (uncontrolled) reactive HiPIMS deposition of stoichiometric dielectric films are: (1) microarcing on the target surfaces at high powers (up to several $kWcm^{-2}$), particularly for the voltage pulses longer than 40 μs, and (2) low deposition rates of the films. To overcome these limitations, the sputter deposition needs to be maintained close to a metallic mode when the compound fraction in the target surface layer is very low, and the position of the RG (reactive gas) inlets in vacuum chamber needs to be optimized to achieve a sufficiently high chemisorption flux of RG particles onto the substrate. Our solution is based on a feedback pulsed RG flow control with a to-substrate RG injection into a dense plasma in front of the sputtered target.

We present and explain a high-rate deposition of two thin-film materials with a great application potential. Strongly thermochromic W-doped VO_2 films with a low semiconductor-to-metal transition temperature of 28 °C were prepared at a relatively low substrate surface temperature of 350 °C (usual temperature is higher than 450 °C) using a controlled deep oscillation magnetron sputtering which is a modified version of HiPIMS with packages (macropulses) of short high-power micropulses. The deposition rate of the highly crystalline thermochromic $V_{0.986}W_{0.014}O_2$ films was 53 nm/min at the target-to-substrate distance of 100 mm [1]. Hard (18 GPa) and highly optically transparent (extinction coefficient of 5×10^{-4} and less than 7×10^{-3} at the wavelength of 550 nm and 300 nm, respectively) HfO_2 films with many potential applications (optics, electronics and nuclear engineering) were prepared at a low substrate surface temperature (less than 165 °C) using a controlled HiPIMS. The deposition rate of the nanocrystalline HfO_2 films with a dominant monoclinic phase was up to 200 nm/min at the target-to-substrate distance of 100 mm [2]. The high deposition rates of the films are explained using a simplified formula and model calculations [3,4].

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Dual-phase nanocomposite coatings based on crystalline ZrN and glassy ZrCu

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Recently, magnetron sputter deposition has been demonstrated to be a suitable deposition technique for the preparation metallic glasses as thin films (TFMGs). Thanks to non-equilibrium conditions of the low-temperature plasma and very high cooling rates on atomic scale at the substrate, TFMGs can be prepared with much wider composition variety and solubility than bulk metallic glasses (BMGs). Moreover, TFMGs have showed properties and characteristics that are superior to BMGs, and metallic and ceramic coatings, e.g., a better balance of ductility and strength. The amorphous structure of TFMGs along with their unique properties also provides a possibility to combine TFMGs with nanocrystalline materials in a heterogenous dual-phase structure. This might allow to overcome the shortcomings of both types of materials and further improve the properties or even discover novel properties based on the synergetic effect of the two phases.

The study focuses on the preparation of dual-phase thin-film materials in the ternary Zr-Cu-N system by reactive magnetron co-sputtering and systematic investigation of their structure and properties. The coatings were deposited in argon-nitrogen gas mixtures using three unbalanced magnetrons equipped with two Zr targets and one Cu target. The magnetron with the Zr targets were operated in dc regimes while that with the Cu target in a high-power impulse regime. All the coatings were deposited onto rotating substrates with rf biasing without external heating. The elemental composition of the coatings was controlled in a very wide composition range.

We have demonstrated that reactive magnetron co-sputtering allows to prepare a new type of the Zr—Cu—N coatings with a nanocomposite structure consisting of two phases, crystalline ZrN and glassy ZrCu. So far, only the nanocomposite Zr—Cu—N coatings based on ZrN and Cu phases have been reported in the literature [1,2]. We show that by varying the process parameters, such as the target power densities, repetition frequency and nitrogen fraction in the gas mixture, we are able to control the elemental composition of the coatings so that the stoichiometry of the two phases remains as much the same as possible and only the volume fraction of the phases is varied. The structure of the as-deposited coatings exhibits a gradual transition from amorphous-like to very fine-grained to nanocrystalline. This transition is reflected in changes in the microstructure and surface morphology and affects the mechanical properties, deformation behavior and corrosion resistance.

Poster 1:

Correlation of Ar substitution by Ne in the plasma and the properties of Cr thin films deposited by HiPIMS-DOMS

In a previous work, the authors have shown that in Deep Oscillation Magnetron Sputtering (DOMS), a variant mode of High-Power Impulse Magnetron Sputtering (HiPIMS), the atomic shadowing mechanism is mostly controlled by the ionization degree of the sputtered material [1]. Thus, at a high ionization degree, dense and compact films can be deposited without the need of high energy particles bombardment. The most straightforward route to achieve high ionization of the sputtered species in HiPIMS is to increase the peak power. However, this also increases the average energy of the sputtered species and brings about energetic bombardment. Partially replacing Ar by Ne in the process gas promotes an increased mean electron energy which increases plasma ionization, as the ionization energy of Ne (21.56 eV) is significantly higher than that of Ar (15.75 eV). In this work, partial substitution of Ar by Ne in the DOMS process gas was investigated as a means to increase the ionization degree of the sputtered species without increasing their average energy.

Cr thin films were deposited by DOMS in pure Ar and mixed Ar + Ne plasmas up to 60 % Ne. Adding Ne to the plasma resulted in 25 % increase in the ions saturation current density (ISCD) as measured by an electrostatic flat probe placed at the substrate location. All the deposited films have a dense and compact columnar microstructure with an almost complete [110] out of the plane preferential orientation. The lattice parameter of the Cr films increased with increasing Ne content in the plasma while their surface roughness decreased from 6 to 1.04 nm. The hardness and young's modulus of the Cr films were evaluated by nanoindentation. All the films properties correlated with the ISCD measured at the substrate location.

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Keywords:
HiPIMS, DOMS, Cr films, Neon, Atomic shadowing

References:

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Surface degradation mechanisms of powder metallurgically manufactured pure Tantalum under erosion-corrosion conditions

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Tantalum (Ta) is a popular choice as a cladding material which is diffusion bonded with the Tungsten (W) core to form a water-cooled spallation target and is used in neutron scattering facilities worldwide. At Rutherford Appleton Laboratory (RAL), this design of target assembly (Ta-W) is currently experiencing rapid deterioration, susceptibly due to erosion-corrosion phenomenon reducing its operational life significantly. This experimental study investigates, previously unreported, aqueous corrosion and erosion-corrosion response of Ta with the help of an aqueous impinging jet erosion-corrosion apparatus. The study examined the effects of varying pH values (4, 7, and 10) and impact angles (30° and 90°) on pure corrosion and erosion-corrosion behaviour of Ta. The results showed that the pure corrosion rate increased with the increase in solution pH irrespective of the impact angle and an opposite trend was revealed for combined erosion-corrosion rate (lowest at pH 10). The reduced erosion-corrosion rate could be attributed to a reduced synergy (antagonistic effect) between erosion and corrosion as the solution became more alkaline. Under all pH values, higher erosion-corrosion rates were obtained for an impact angle of 30° compared to the normal impact angle (90°). This corresponds well with the earlier work on pure erosion studies in which Ta demonstrated a ductile erosion behaviour (higher mass erosion at oblique impact). The results also indicated that plastic deformation and micro-cutting were the primary material removal mechanisms which constituted the mechanical component irrespective of the corrosive solution (pH 4, 7, and 10). Dissolution of the deformed material, micro-pitting along with mechanical removal of passive layers constituted the corrosion mechanisms.

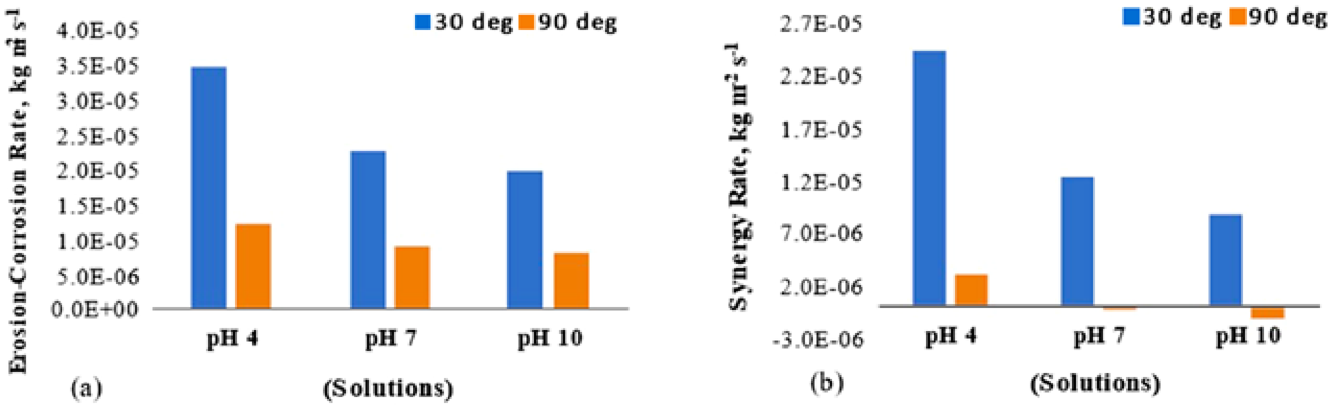

Fig: Bar charts showing effect of pH variation of pH (4, 7, and 10) and impact angles (30°, 90°) on (a) Erosion-corrosion rate and (b) Synergy rate.

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Poster 3:

Improved platinum thin film quality using bipolar HiPIMS deposition

Platinum thin film is widely being used as an electrode in micro and nanoelectronics devices due to its high electrical conductivity and low chemical affinity. Low-temperature grown Pt film usually forms defects and cracks when exposed to high temperatures. In this study, thin platinum films were grown by bipolar high-power impulse magnetron sputtering (bi-HiPIMS). The film properties were compared to films grown by conventional dc magnetron sputtering (dcMS) and radio frequency (RF) sputtering at similar conditions.
It was found the film density, surface roughness, and electrical conductivity of HiPIMS-grown film are superior to the films grown by dcMS and RF. In addition, HiPIMS-deposited films show smoother surfaces and fewer defects after annealing at 600 °C compared to their counterparts. However, the deposition rate is found to be lower for sputtering by HiPIMS than for dcMS and RF.

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Poster 4:

Sputter Deposition of p-type Thin Films for Partner Layers in n-type Cadmium Telluride Solar Cells

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Conventionally, cadmium telluride photovoltaic devices consist of p-type CdTe absorber layers. The use of n-type CdTe absorber layers offer the potential advantages of higher stable doping densities and lower back contact barriers. The n-type doping of CdTe thin films has been studied elsewhere, utilising both post-growth [1] and in-situ [2] mechanisms. However, very little work has been done regarding the architecture of a full photovoltaic device in relation to an n-type CdTe absorber layer. As a result, no results of potential p-type partner layers for n-type CdTe thin film solar cells have been reported within literature. This work explores the use of various p-type partner layers for n-type CdTe solar cells, reporting on both individual film characteristics and full device performance. The p-type partner layers investigated include ZnTe, PEDOT:PSS, CuI, CuSCN and NiO, along with n-type/intrinsic layers including CdSe and TiO^2. These layers were predominantly deposited via magnetron sputtering or else solution processing methods.

Of the sputtered p-type partner layers investigated, ZnTe showed the greatest potential with a device efficiency of 1.5% in both substrate and superstrate configurations. Post-growth doping of ZnTe thin films with Cu resulted in the carrier concentration increasing from ~10^{16} cm^{-3} with no Cu content to ~10^{20} cm^{-3} with 4 wt% Cu content. Devices with NiO partner layers showed weaker performance but film characteristics revealed several insights. Firstly, the optical transmission of the films was found to increase with increasing substrate temperature during growth up to 200°C. Secondly, the band gap of the films was found to decrease with increasing oxygen content during growth, from 3.26 eV with 0% oxygen content to 2.97 eV with 20% oxygen content (relative to argon process gas). Finally, the post-deposition annealing of the films was found to have little-to-no effect on optical or electric properties. Overall, ZnTe offers an ideal p-type partner layer for n-CdTe solar cells, whereas CdSe may provide higher performance as an intrinsic layer.

Poster 5:

Influence of pulse duration on plasma chemistry and thin film growth of plasmonic Titanium Nitride deposited by HIPIMS

Titanium Nitride was deposited utilising constant current HIPIMS whilst the pulse duration was varied, this allows for the investigation of plasma parameters as well as the texture of the thin films and enabled to optimise the optical properties of the thin films. The pulse durations investigated were between 40 – 200 µs. During the investigations the peak current density reached values of 0.6 Acm^{-2} on a 100 mm diameter cathode. Optical Emission Spectroscopy (OES) shows that the plasma chemistry develops from gas dominated at pulse duration of 40 µs to metal dominated at 60 µs and above. Analysis of OES data shows there is a relative increase in Titanium neutral emission within the plasma with increasing pulse durations. However, Ti III emission intensity shows the strongest relative increase. Despite the constant current, there is shown to be a continuous increase of emission from metal ions and neutrals relative to Argon neutrals within the plasma as the pulse duration is increased. Films were deposited for each investigated pulse duration, pole figures conducted on said films suggest a strong (111) crystallographic orientation for the longer pulse durations. The Titanium Nitride films have been shown to be plasmonically active when exposed to radiation within the visible light spectrum. The films deposited at a temperature of 300 °C exhibited excellent plasmonic activity with optical losses on par with DC magnetron sputtered films deposited at 600 °C.

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Poster 6:

Improving tribocorrosion resistance of a medical grade CoCrMo alloy by the novel HIPIMS nitriding technique

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Research has indicated that tribocorrosion resistance (resistance to the coaction of wear and corrosion simultaneously) demands a higher precedence while choosing materials for prosthetic joints and medical implants. Better individual wear and corrosion resistance of a material does not guarantee better tribocorrosion resistance as complex tribo-corrosive mechanisms reign. Most of the materials do not inherent favourable attributes and needs engineered surfaces to improve their longevity. With this foresight, recently, a novel nitriding process based on the plasma generated by the High Power Impulse Magnetron Sputtering (HIPIMS) technique has been developed.

HIPIMS Low pressure Plasma Nitriding (HLPN) technique has successfully improved mechanical properties, wear and corrosion resistance of medical grade (F75) CoCrMo alloy. Current work analyses the sliding wear response of these nitrided alloys carried out in a corrosive simulated body fluid environment (Hank's solution) with simultaneous corrosion monitoring. Under the Open Circuit Potentials (OCP), both nitrided and the untreated specimens exhibited a near similar Sliding-Wear-Corrosion coefficient (K_{SWC}) values (6.52×10^{-15} and 3.95×10^{-15} respectively). However, the benefits of HLPN were clearly visible under the accelerated corrosion (anodic potentials) and passivating conditions where the nitrided specimens exhibited an order of magnitude higher resistance to wear ($K_{SWC} = 6.41 \times 10^{-15}$ and 4.29×10^{-15} respectively) as compared to the untreated alloy ($K_{SWC} = 3.40 \times 10^{-14}$). This work focuses on analysing the influence of microstructure on the significant improvements in tribocorrosion performance of nitrided specimens observed.

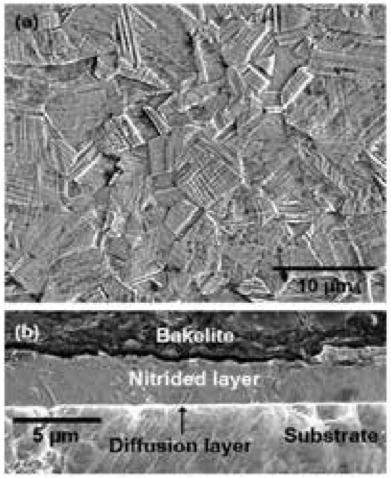

Fig.1: Microstructure (a) nitrided surface in plan view (b) cross-sectional view.

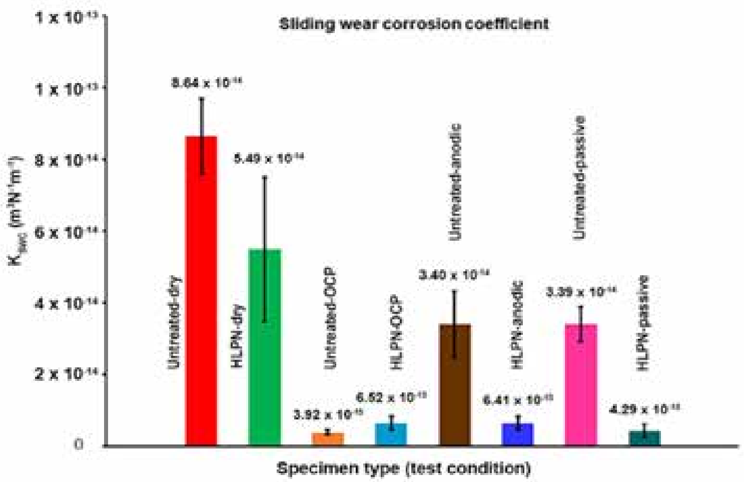

Fig.2: Sliding Wear Corrosion coefficients (K_{swc}) calculated

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Poster 7:

Low-Pressure Plasma Nitriding of Medical Grade AlloyUsing HIPIMS Discharge

CoCrMo alloys were plasma nitrided using both HIPIMS and DC discharge to compare the quality and productivity of the process. In this work, phase and layer composition, hardness, tribological and corrosion resistance of both samples have been investigated. Results revealed that the HIPIMS develops a mix nitrided layer (Co_4N + Co_4N-$Co_{2-3}N$) two times thicker than that of DC plasma nitrided (DCPN) (only Co4N-Co2-3N). Based on surface hardness analysis HIPIMS plasma nitrided (HPN) showed a significant increase in hardness value (23 GPa) as compared to DCPN (20 GPa) and the untreated one (~8 GPa). Surprisingly, both specimens showed similar tribological properties. However, HPN showed superior corrosion resistance (i_{Corr} = 10^{-5} mA) as compared to DCPN, 10^{-4} mA.

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Poster 8:

Realtime control of composition of CrAlYN/CrN coatings deposited by high power impulse magnetron sputtering

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The objective of this study was to produce CrAlYN/CrN nanoscale multilayer coatings with reproducible composition by controlling the optical emission line of Cr I neutral excited atoms at 425.4 nm while changing the unbalancing coil current from 0 A to 8 A. The coatings were deposited using an industrial scale high power impulse magnetron sputtering (HIPIMS) enabled coating machine at Sheffield Hallam University. Two HIPIMS and two dc-magnetron sputtering sources were used for the reactive deposition. The average power was kept constant at 8 kW on all four cathodes. The duration of the coating process was kept constant at 4.5 hr. Optical Emission Spectra (OES) were recorded during the deposition of the coatings using a Czerny-Turner type monochromator (Triax 320) with a quartz optical fibre attached to a telescopic lens feedthrough attached to the chamber wall. The lens collimated the light from the racetrack of the cathode and focused it onto the quartz fibre. A Speedflo plasma emission monitoring system was operated in "sensor" mode i.e. the N_2 gas flow was adjusted to keep the observed emission intensity of Cr I neutral excited atoms from one cathode at a desired set point value throughout the deposition process. Various set point values were used to demonstrate that the coating composition can be controlled by controlling the optical emission intensity of the Cr I neutral excited atoms. To choose the sensor set point values to deposit stoichiometric coatings with compositional reproducibility, the hysteresis in observed sensor signal for Cr I while varying the nitrogen flow from 0 to 300 sccm and back to 0 was studied for all coil currents from 0 A to 8 A. The EDX analysis showed that the coatings were under-stoichiometric in nitrogen with almost no change in composition and stoichiometry. X-ray pole figure measurements showed that these coatings had (111) and (220) preferred orientations depending on the coil current. Scanning electron microscopy analysis revealed that the microstructure varied from faceted to dense while varying the coil current from 0 A to 8 A.

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Poster 9:

Characterizations and drill performance of AlCrTiN coatings deposited with various target power and bias voltage by high power impulse magnetron sputtering.

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In recent years, the production capacity of printed circuit boards (PCBs) has increased significantly resulting in an increase in the demand for micro drills. Therefore, it is particularly important to develop high-lifetime drills, and the extended service life of drills can be achieved via coatings technology. Optimum mechanical properties of hard coating can be achieved by high power pulsed magnetron sputtering (HiPIMS). In this study, AlCrTiN coatings were prepared via HiPIMS with four $Al_{70}Cr_{30}$ targets and two Ti targets, with a focus on the effects of target power (AlCr: 3 to 5 kW, Ti: 1 to 5 kW) and substrate bias voltage (from —30 to —90 V) on the deposition rate, microstructure, crystal orientation, residual stress, and mechanical properties of the coatings. FE-SEM revealed that increasing the target power or bias voltage decreased the grain size of the coatings and the structure became denser. By increasing the Ti content in AlCrTiN coating, XRD revealed the preferred orientation change from (100) to (111). When the output power is the maximum, the hardness value is 2931 Hv, the adhesion force is 30.8 N, and the wear rate can reach the best result of 5.94 $\times 10^{-7} mm^3 N^{-1} m^{-1}$. The highest hardness (3753 Hv) and highest adhesion force (48.42 N) were obtained by increasing the bias voltage (—75 V). The drill test results showed better wear resistance and useful lifetime than TiAlN coating for PCBs application.

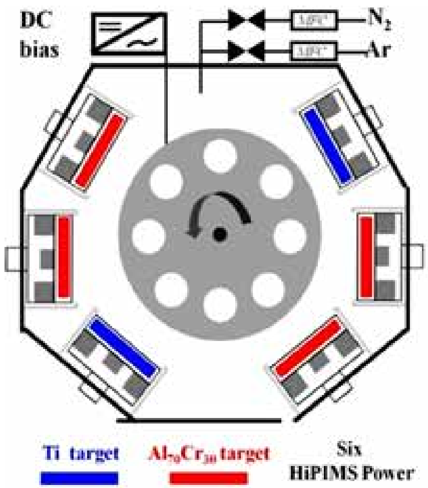

Fig a: Schematic cross section of the coating

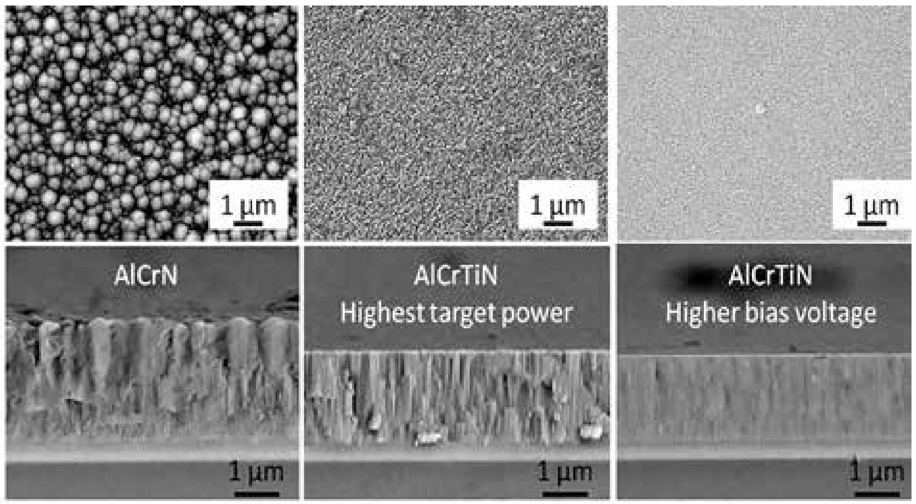

Fig b: SEM plan view and cross section images from the various coating

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Poster 10:

Deposition of Ga_2O_3 and $ZnGa_2O_4$ thin films by liquid metal target sputtering

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Reference:
[1] M. Zubkins et al. Vacuum 209 (2023) 111789.

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This study reports on the deposition of amorphous and crystalline thin films of Ga_2O_3 [1] and ZnGa2O4 by reactive pulsed direct current magnetron sputtering from a liquid gallium target onto fused (f-) quartz and c plane (c-) sapphire substrates, where the temperature of the substrate is varied from room temperature (RT) to 800 °C. A Zn target was co-sputtered next to the Ga target to produce $ZnGa_2O_4$ films. The composition was controlled by the sputtering power of the Zn target, and the process was monitored by plasma optical emission spectroscopy. The static deposition rate of Ga_2O_3 (up to 37 nm/min at RT on f-quartz and 5 nm/min at 800 °C on c-sapphire) is two to five times higher than the data given in the literature for radio frequency sputtering. Deposited onto unheated substrates, the films are X-ray amorphous. Well-defined X-ray diffraction peaks of β-Ga_2O_3 start to appear at a substrate temperature of 500 °C. Films grown on c-sapphire at temperatures above 600 °C are epitaxial. However, the high rocking curve full width at half maximum values of ≈2.4–2.5° are indicative of the presence of defects. A dense and void-free microstructure is observed in electron microscopy images. Composition analysis show stoichiometry close to Ga_2O_3 and no traces of impurities. The optical properties of low absorptance (<1%) in the visible range and an optical band gap of approximately 5 eV are consistent with the data in the literature for Ga_2O_3 films produced by other deposition methods. $ZnGa_2O_4$ films start to crystallise around 200 °C according to XRD. The shift of an optical band gap with the Zn concentration is observed.

Exhibitor list
Thirteenth International Conference on HIPIMS

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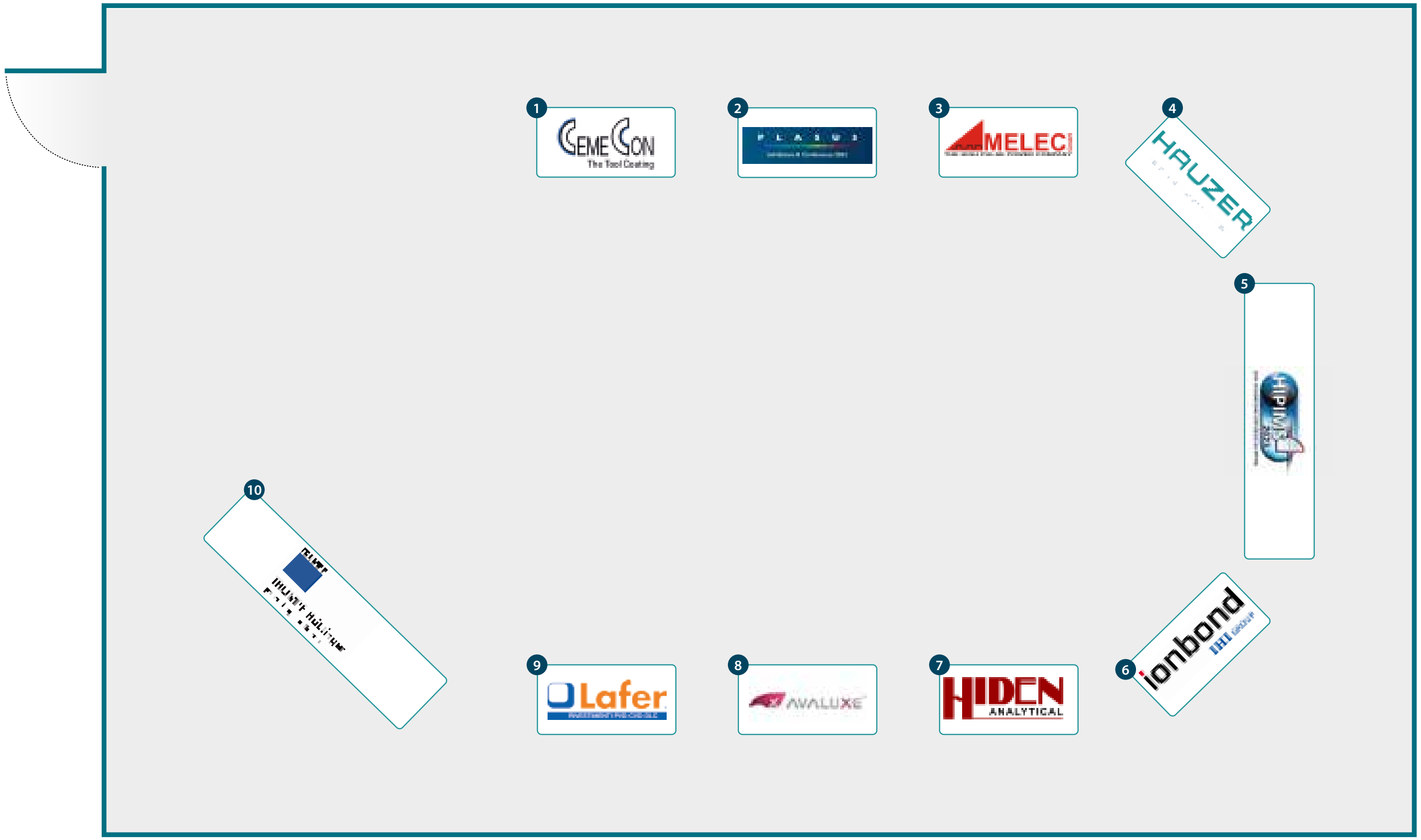
9. Lafer S.p.A
10. TRUMPF Huettinger Electronic Sp. z o.o.



Notes