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ST – HIPIMS – Conference

International Conference on Sputter Technology &
10th International Conference on Fundamentals and Applications of HIPIMS

BOOK OF ABSTRACTS

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Preface

Dear colleagues,

we are celebrating the 10th anniversary of the International Conference on Fundamentals and Applications of HIPIMS this year in Braunschweig. We are looking back to a great evolution of one of the greatest developments in sputtering that was ignited at the end of the last century. From the Sheffield HIPIMS days the HIPIMS Conference was born with the vision to grow the community, advance technological development, and expand the fundamental understanding as well as the industrial implementation. Looking back, we can see that the joint activities from Sheffield Hallam University, Fraunhofer Institute for Surface Engineering and Thin Films IST and the Network of Competence Industrial Plasma Surface Technology INPLAS play a key part in maturing this technology. Today HIPIMS has become a well known technology and is used in several industrial applications and production processes. Besides sputtering, also arc technology has strongly benefitted from developments in recent years, especially the development of advanced power supplies and plasma diagnostics suited for high plasma densities. With the general development in PVD technology, several requests from industrial partners, and the more general technological approach in commercial applications, the spectrum of sputter technologies covered up to now in the HIPIMS conference will be broadened. Therefore, besides the 10th anniversary we are celebrating the launch of the International Conference on Sputter Technology which will be held in Braunschweig biannually jointly with the HIPIMS conference. This marriage is also expressed in the conference's short name ST-HIPIMS Conference. This year's conference will link latest advances in HIPIMS and sputtering technology. The maturity of the HIPIMS technology is reflected by one session presenting industrial coating systems, process control and production processes. Nevertheless the fundamental aspects will be treated as well as latest coating developments. Additionally, sputtering technology will be given a more general focus with high level contributions on ion beam sputtering, gas flow sputtering, and more general topics like surface integrated sensor systems or cyber-physical production systems in surface technology. To strengthen the active interaction between the participants different occasions are available: a guided poster session, face-to face discussions and match making at the industrial exhibition, active discussion at the new breakfast discussion forum, or the networking event with the awards ceremony.

We are looking forward to an amazing event and your active contribution to make this years ST-HIPIMS Conference a unique and successful event!

Dr. Ralf Bandorf and Prof. A. Ehiasarian
Conference Chairman and Co-Chairman of
ST-HIPIMS-Conference 2019

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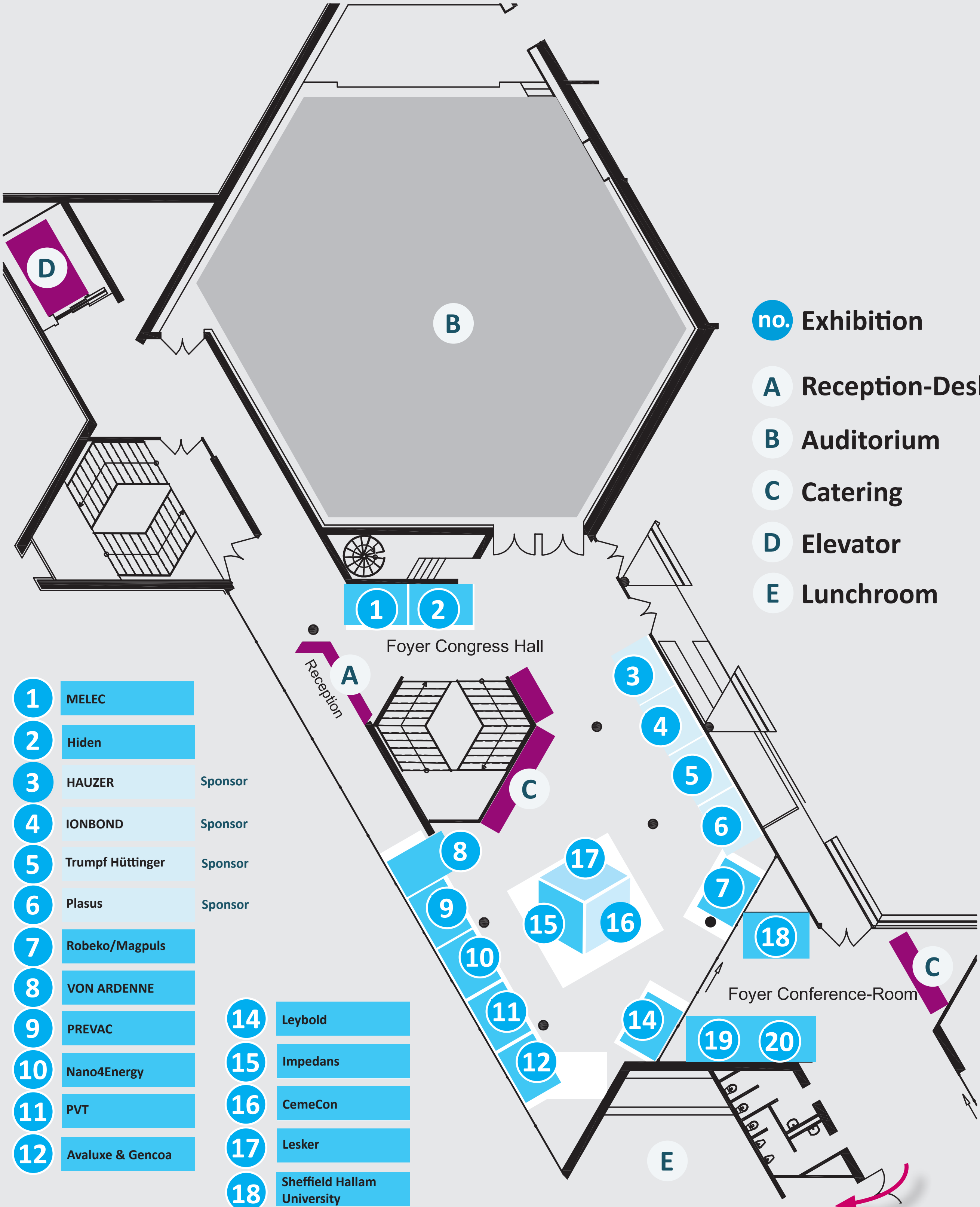
HIPIMS 2019
10th International Conference on HIPIMS

Sputter Technology 2019
International Conference on Sputter Technology



no. Exhibition

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20 Fraunhofer Institute for Surface Engineering and Thin Films
Braunschweig, Germany
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ORAL PRESENTATION

ABSTRACT 1

Tribological properties of low pressure plasma nitrided CoCrMo alloy using HIPIMS discharge

KRISHNANAND SHUKLA*, ARUNPRABHU A. SUGUMARAN[1], IMRAN KHAN[2], A. P. EHIASARIAN[1], P. EH HOVESPIAN[1]

National HIPIMS Technology Centre, Materials and Engineering Research Institute, Sheffield Hallam University, UK S1 1WB.

***CORRESPONDING AUTHOR**
E-MAIL: KRISHNANAND.SHUKLA@STUDENT.SHU.AC.UK

CoCrMo is a biomedical grade alloy which is widely used in the manufacturing of orthopaedic implants such as hip and knee replacement joints because it has high hardness, better corrosion resistance, and excellent biocompatibility. However, the major concern is the release of toxic metal ions due to corrosion and wear of the alloy, which causes an allergic reaction in the human body. Over the years various surface modification techniques including nitriding have been used to improve the performance of CoCrMo (F75) alloy.
In the current work a new low pressure plasma nitriding process is described. Unlike convetional plasma nitriding the process utilised HIPIMS discharge sustained on one Cr target at low power to further enhance the ionisation of the gas in the vacuum chamber and avoid coating deposition.
The nitriding of CoCrMo alloy has been carried out in a wide range of nitriding voltages (from -500 V to -1100 V) at 4000C. The chemical and phase composition of the nitrided layer has been studied by various advanced surface analyses techniques.
The X-ray diffraction data of all nitrided samples revealed the formation of expanded austenite (ϒN) phase.
Texture analyses revealed that at lower nitriding voltages (UN=-700V) the predominant crystallographic orientation of the compound layer is (200) whereas at higher voltages (UN=-900V, -1100V) the layer develops mixed (111) and (200) texture. For samples nitrided at lower, UN=- 500 V substrate bias, diffraction peaks for CrN/NbN and Cr2N were also observed due to the deposition of target materials (Cr and Nb). However, no coating deposition on the substrate surface was observed at higher (UN=700V and higher) bias voltages due to sufficient re-sputtering effect. The results obtained from secondary ion mass spectrometry (SIMS) and glow discharge optical emission spectroscopy (GDOES) depth profiling have showed that the depth of nitriding increased from approximately 0.7 µm at -500 V to 6 µm at -1100 V. In pin-on-disc tribological test nitrided samples showed low coefficients of friction (COF) in the range of µ= 0.6 to µ= 0.7, compared to µ= 0.8 recorded for un-treated substrate. The wear coefficients (Kc) were found to be between 1.79x10-15 m3N-1m-1 (UN=-700 V) and 4.62x10-15 m3N-1m-1 (UN=-1100 V) which were one order of magnitude lower as compared to the untreated substrate, 6x10-14 m3N-1m-1. The nanohardness (H) of nitrided samples significantly increased by a factor of 3 (22 GPa at Ub=-1100 V) as compared to the untreated substrate, 6 GPa showing the high efficiency of the process.

ABSTRACT 2

High-performance thermochromic VO2-based coatings prepared on glass by a low-temperature (330 °C) scalable technique

J. VLČEK, D. KOLENATÝ, T. BÁRTA, J. REZEK, J. HOUŠKA, S. HAVIAR

Department of Physics and NTIS – European Centre of Excellence, University of West Bohemia, Univerzitni 8, 306 14 Plzen, Czech Republic

***CORRESPONDING AUTHOR**
E-MAIL: VLCEK@KFY.ZCU.CZ

Thermochromic VO2-based coatings were prepared on soda-lime glass by a low-temperature scalable deposition technique. This deposition technique is based on reactive high-power impulse magnetron sputtering with a pulsed O2 flow control [1] allowing us to prepare crystalline VO2 layers of the correct stoichiometry under highly industry-friendly deposition conditions: without any substrate bias at a low substrate temperature of 330 °C. Simultaneous

doping of VO2 by W (resulting in a V1-xWxO2 composition with x = 0.018 in this work) was performed to reduce the semiconductor-to-metal transition temperature to 20 °C. ZrO2 antireflection layers both below and above the thermochromic V0.982W0.018O2 layers were deposited at a low substrate temperature (< 100 °C). A coating design utilizing a second-order interference on the ZrO2 layers [2] was applied to increase both the luminous transmittance, Tlum, and the modulation of the solar transmittance, T_{sol}. The crystalline structure of the bottom ZrO2 layer further improved the VO2 crystallinity and the process reproducibility. The top ZrO2 layer provided the mechanical protection and environmental stability of the V0.982W0.018O2 layers. The ZrO2/V0.982W0.018O2/ZrO2 coatings exhibited Tlum up to 60% at $\triangle T_{sol}$ close to 6% for a V0.982W0.018O2 thickness of 45 nm, and Tlum up to 50% at $\triangle T_{sol}$ above 10% for a V0.982W0.018O2 thickness of 69 nm. The results are important for a low-temperature fabrication of high-performance durable thermochromic VO2-based coatings for smart-window applications.

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Abstract 3

Super thin CrN coatings with high emissivity on barium fluoride to investigate heat flux of bubble formation

A. Martin*[1], H. Scheerer, T. Engler, M. Oechsner

Zentrum für Konstruktionswerkstoffe, Staatliche Materialprüfanstalt Darmstadt, Fachgebiet und Institut für Werkstoffe, Grafenstraße 2, 64283 Darmstadt

***Corresponding author**
E-mail: martin@mpa-ifw.tu-darmstadt.de

In many technical processes such as for example microfluidic, Lab-on-a-chip technology and cooling technology the bubble formation properties on the surfaces in fluids play an important role. Wetting as well as dewetting processes are mutual and closely connected to local impulse, heat and mass transport processes. These nano- and microscopic processes and their interactions determine in many cases significantly the efficiency of the whole technical process as well as the quality of the resulting products. Of high interest is the three phase contact line (fluid, gas, solid). Therefore, a complex setup is developed to determine small dimensional heat transfer processes in high resolution. The work is being done in cooperation with the Institute of Technical Thermodynamic Technical University Darmstadt and Airbus-Defence and Space for experiments on the International Space Station (ISS) and in parabolic flights. The main component is a heater made of a PVD bilayer on a barium fluoride single crystal substrate.
On top a Cr coating is working as an active heating element. To investigate the three phase contact line on the back side to the crystal a very thin, low electrical resistance coating with high emissivity (wavelength 8 -10 µm) is needed.
A combination of High Power Impulse Magnetron Sputtering (HiPIMS) and Direct Current Magnetron Sputtering (DCMS) technology with different bias voltage, nitrogen proportion and thickness of the coating was used to investigate the influence of these parameters on the emissivity and lattice orientation of the coating. Thickness determination (Confocal microscope), Scanning electron microscope (SEM), X-ray diffraction (XRD) combined with emissivity analyses was used to find an optimum of all essentially parameters. A 400 nm thin CrN coating with strong (111) crystal lattice orientation was found the have an emissivity of 0.8 (temperature range 30 -70 °C) with low electrical resistance with specifically designed electrical contact allow to heat only with the Cr layer on top (Figure 1, Figure 2). First IR camera images made under weightlessness in parabolic flights show due to high resolution the functionality of the found coating parameters. In May 2019 the experimental setup will fly to ISS.

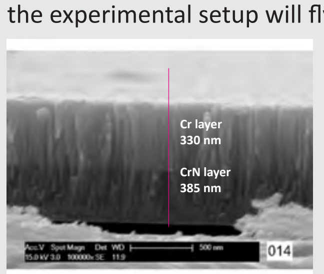

Figure 1:
SEM image of the Cr/CrN bilayer with high emissivity CrN layer

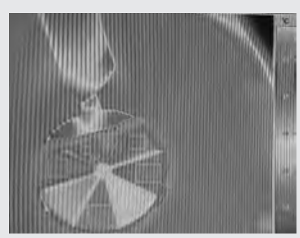

Figure 2:
IR- Image of different coatings at the same temperature. The emissivity can be determined indirectly.

Abstract 4

Advanced HIPIMS coatings through Kick pulse technology

Jason Hrebik*

Kurt J. Lesker Company, 15/16 Burgess road, TN35 4NR Hastings, UK

***Corresponding author**
E-mail: jasonh@lesker.com

HIPIMS coating technology has been rapidly growing over the past few years due to the availability of R&D scale supply offerings. This has resulted in many new breakthroughs in application enhancement, production scalability, and efficiency. The number of applications where HIPIMS is now considered is also advancing. Breakthroughs in HIPIMS controllability have enabled researchers to find a variety of ideal operating parameter sets for various performance requirements. One of the most significant technical advances is a reverse positive kick pulse. This option provides a significant variable for driving out film stress in HIPIMS applications and increasing yield rates, which have been a major downside to HIPIMS in the past. These advances open up new possibilities for the technology and the enhancement of many thin film applications. This presentation will highlight examples of these applications along with the advantages associated with HIPIMS and the Kick pulse technology. It will show how these advances can be scaled to larger scale production applications and provide examples of what enhancements can be expected.

Abstract 5

High deposition rate films prepared by reactive HiPIMS

P. Mareš[1], M. Dubau[1], A. Marek[1], J. Vyskočil[1], J Čapek[2], T. Kozák[2]

[1]HVM Plasma spol. s r.o., Na Hutmance [2] Praha, 518 00, Czech Republic [2]NTIS - New Technologies for the Information Society, Faculty of Applied Sciences, Technicka 8, Pilsen, 301 00, Czech Republic

***Corresponding author**
E-mail: pavel.mares@hvm.cz

High power impulse magnetron sputtering (HiPIMS) is characterized by a very high power density at the target during each pulse with very low duty cycles in order to allow cooling of the target. High fraction of sputtered target material is ionized during the pulse which allows the preparation of hard, dense and defect-free coatings. However, one of the greatest disadvantages of the HiPIMS technology is its lower deposition rate due to the return of ionized target material back to the target. This limitation hinders wide application of HiPIMS technology in the industry [1].
Reactive magnetron sputtering from metal target is a well-established technique to deposit compound films at different composition by controlling the reactive gas flow. Target poisoning and resulting reduced sputtering rate influence the deposition process stability and give rise to hysteresis effects [2,3]. Recently, a suppression of hysteresis effects using reactive HiPIMS was shown [4] and explained [5] by a mathematical model. One of the fundamental results of this model shows that in some cases reactive HiPIMS can achieve higher deposition rates compared to middle frequency pulsed dc reactive sputtering due to the lower rate of target poisoning.
The main aim of this work is to confirm this prediction experimentally and find compound films exhibiting higher deposition rate at HiPIMS condition than in more common middle frequency pulsed DC sputtering techniques.
To confirm these model predictions experimentally we had to choose proper materials, where we can demonstrate the increase of deposition rate at HiPIMS conditions.
The simulations were performed in Monte-Carlo simulation programs SRIM [6] and SDTrimSP [7].
We searched for materials which have sputtering yield from poisoned target very low in comparison to clean metal target. In this case deposition can benefit from lower rate of target poisoning at HiPIMS conditions.
We tested Al_2O_3, Cr_2O_3, TiO_2, ZrO_2, HfO_2, Nb_2O_5 and Ta_2O_5. The best results were achievedfor Nb_2O_5 and Ta_2O_5. The experiments were performed in laboratory system Flexilab of our own production [4] equipped with three magnetron cathodes with targets of 2” in diameter. Two of them were used in dual bipolar regime powered by 2kW ADL power supply operated at constant power 200 W in combination with a MAGPULS SB 1000/100/200 bipolar pulsing unit operated from middle frequency pulsed DC conditions (duty cycle 45:5 µs) up to HiPIMS

conditions (duty cycle 25:725 µs). The current density at sputtering targets during a pulse increased typically from 0.02 Acm-2 at middle frequency pulsed DC conditions up to 2 Acm-2 at HiPIMS conditions. Partial pressures of gases were measured by a quadrupole mass spectrometer Pfeiffer Vacuum SPM 220 calibrated by capacitance gauge. Thickness of deposited coatings was measured by ellipsometry (Alpha-SE ellipsometer, J. A. Woollam), confocal microscopy (LEXT OLS4000, Olympus), XRF (Fischerscope X-Ray XDLM 237, Helmut Fischer GmbH) and by mechanical profilometry (Dektak 8 Stylus Profiler, Veeco). In some cases the mass of the targets before and after deposition was measured on the analytical weight (Sartorius type 2492). The chemical compostion of the films was measured by SEM (JEOL JSM 6460 LA). Mechanical properties (Picodentor HM 500, Helmut Fischer GmbH) and optical properties (Alpha-SE ellipsometer, J. A. Woollam) were also analyzed. We have confirmed that high deposition rate of Ta2O5 and Nb2O5 films can be achieved using HiPIMS with maintaining mechanical and optical properties. Therefore, reactive HiPIMS seems to be a suitable method for production of some types of compound films.

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[6] SRIM, http://www.srim.org/
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Abstract 6

Comparison of Langmuir probe and laser Thomson scattering for plasma density and electron temperature measurements in HiPIMS plasma

Peter J. Ryan*[1], James W. Bradley, Mark D. Bowden

[1]Department of Electrical Engineering and Electronics, University of Liverpool, Liverpool L69 3GJ, United Kingdom

***Corresponding author**
E-mail: ryanp@liverpool.ac.uk

The plasma dynamics of HiPIMS has been investigated extensively with the aim of better understanding the creation and transport of metal ions to the substrate so that the deposition process can be optimised [1]. Electron property measurements are fundamental to any physics investigation because metal ions are produced predominately by electron impact ionisation, and magnetised electrons guide ions through ambipolar diffusion to the substrate in an unbalanced magnetron configuration. Electron density and temperature are typically measured using the Langmuir probe technique but most studies are restricted to locations outside of the magnetic trap, e.g. [2, and therein]. This is both to avoid perturbing the main ionisation zone of the discharge, and because the interpretation of the probe data is simpler in regions where the magnetic field strength is lower. In this contribution we report the first comparison of plasma density and electron temperature measurements in HiPIMS plasma made by Langmuir probe and laser Thomson scattering. The interpretation of the Thomson data is unambiguous and unaffected by magnetic fields, and so reliable electron properties can be obtained with excellent spatial (3 mm) and temporal resolution (5 µs). Measurements were performed (non-simultaneously) at two positions within the plasma; in the low B-field region on discharge axis and in the high B-field region (~33 mT) of the magnetic trap, for peak power densities of 450 Wcm−2 and 900 Wcm−2 respectively. The maximum plasma densities and temperatures were 6.9×1019 m−3 and 3.7 eV in the pulse-on time, and values decayed to 4.5×1017 m−3 and 0.1 eV at times up to 350 µs from the pulse initiation. The results indicate that although intrusive, the Langmuir probe can provide a reasonable indication of electron properties in different regions of electron magnetisation within the discharge. Further Thomson scattering measurements in the magnetic trap are planned to investigate the spatial distribution of electron density and temperature. The data will be correlated with optical emission spectroscopy to obtain information about species abundance.

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Abstract 7

HIPIMS/UBM PVD coating equipment designed to coat universal sized broaches

W.-D. Münz, R. Klink, D. Aleksic

Arthur Klink GmbH, Steinenlandstr., Pforzheim, Germany

***Corresponding author:**
E-mail: w-dm@gmx.at

Broaches are typically dimensioned in a diameter range of 10 to 500 mm and 200 up to 2500 mm in length. In general this broad range of dimensions represents a tremendous challenge towards the layout of sputter geometry. Especially varying distances of tool to cathode surfaces demand particular care to succeed in dense coating structures. This is particularly valid in large coating chambers, if the tools are placed e.g. vertically parallel to the cathodes. In case of differently long tools the target yield becomes an important aspect especially if short tools in small numbers should be coated alternatively to long tools. Long cathodes would blow waste material into the empty chamber space consequently. The cathodes involved are mounted to two doors of an octagonal vacuum chamber. The height reaches approx. 4 m. Each door carries one 2.5 m long UBM with a width of 20 cm. To achieve an identical active length for HIPIMS three 0.9 m long cathodes 15 cm in width were lined up. The plasma regimes generated are slightly overlapping to realize the same total length as the UBMs. The UBMs are powered by 3 x 20 kW each and the HIPIMS cathodes with 20 kW per cathode. HIPIMS is operated with – 1000 V, pulse widthes 80 to 120 µsec and a pulse repetition frequency of 500 Hz. Substrate biasing is achieved by a synchronized HIPIMS supply. The UBMs are furnished with full length targets. To address the different tool length problem the targets are magnetically segmented into two sections by using two independent movable magnet arrays. One array covering 2/3 and the second array the rest 1/3 of the target. Withdrawing one of the arrays by approx. 15 cm from the target rear side means no ignition possible in this target regime. Keeping the magnets close to the target rear allows ignition. Three independent tool length categories can be served reasonably. Dependent on the tool size the UBMs are fired either with 20 or 40 kW. The three HIPIMS cathodes are opposing these target segments accordingly. OEM analysis monitors the plasma distribution.
An important issue in the design of the PVD process described here is the combined application of HIPIMS and Unbalanced Magnetron cathodes. HIPIMS is used to generate ion impantation of Cr to achieve excellent coating adhesion. Furthermore HIPIMS process increases the ionization level in the chamber HIPIMS deposited CrN together with the UBM deposited TiAlN/TiAlVN increases the quality of the growing films. Beginning with conventional TiN a variety of multilayer coatings have been deposited and their properties have been explored.

Abstract 8

Influence of high voltage discharge on the plasma nitrided zone

C. Kipp*[1], P. Kaestner, G. Bräuer

Institute for Surface Technology, IOT Technische Universität Braunschweig, Bienroder Weg 54E, 38108 Braunschweig, Germany

***Corresponding author**
E-mail: p.kaestner@tu-bs.de

An up-to-date DC PVD power supply, with its wide range of plasma parameters, was used for the well-established plasma nitriding process for surface hardening of steel and stainless steel. Due to its stable arc-management, it was possible to increase the voltage from a typical value of 500 V up to 1000 V. The benefit of high-voltage on long-term plasma nitriding processes (16h) is a greater thickness of the compound layer.

Between 500 V and 800 V the compound layer growth increased from 8 µm to 12 µm. The nitriding depth also showed a slight increase. A greater effect, however, was achieved in short-term plasma nitriding processes at higher voltage

Figure 1: Development of compound layer thickness with increasing voltage for short time plasma nitriding processes, 2 hours on the left and 0.5 hours on the right constant parameters: pressure 300 Pa; temperature 565°C; pulse-to-pause ratio 100 µs: 400 µs; atmosphere 80% nitrogen, 20% hydrogen

Figure 1 shows that by increasing the voltage from 500 V to 800 V a significant growth of the compound layer thickness was observed. An increase from 1.9 µm to 8.8 µm was found after 2 hours treatment time and an increase from no compound layer to 5.2 µm after 0.5 hours. A similar compound layer thickness could be produced after 2 hours at 800 V instead of 16 hours at 500 V. In addition, the nitriding depth increased significantly in the short-term process at higher voltage. The thickness rose from 65 µm (500V) to 90 µm (800V) after 2 h and from 10 µm (500V) to 40 µm (800V) after 0.5 h. Therefore, the high voltage has a major influence on the growth of the compound layer and the nitriding depth in short-term plasma nitriding processes. The talk gives a brief introduction to plasma diffusion treatment and then shows the benefit of higher voltage on the process in detail.

Abstract 9

Combined control of ionization and stoichiometry in reactive highly ionized processes for production lines

T. Schütte, P. Neiss[*1,] Julius Rieke, Günter Bräuer[2,] Holger Gerdes, Ralf Bandorf, Günter Bräuer[3]

*[*1]PLASUS GmbH, Mering, Germany*

[2]IOT Braunschweig, Braunschweig, Germany

[3]Fraunhofer IST, Braunschweig, Germany

***Corresponding author**
E-mail: schuette@plasus.de

While reactive high-density plasma processes, like HIPIMS, are more and more adapted in industrial plasma applications, a reliable and stable control technique is crucial for securing deposition parametersin production processes. In reactive HIPIMS applications, the chemical composition (i.e. layerstoichiometry) and the film physical properties like density are depending on the gas composition of metal to reactive species and the degree of ionization. However, these parameters are influencing each other in highly ionized plasmas and thus, a single control on either gas flow or peak current is not sufficient to run a stable production process. Especially with progressing target erosion, the work point will shift and in the worst case run out of any tolerance without proper control.
By combining the measurement of pulse current and pulse voltage with the spectroscopic plasma monitoring technique, independent control of degree of ionization and reactive gas flow can be realized, thus securing chemical composition and physical film properties at the same time. While the spectroscopic data reveals mainly information about the gas composition, the analysis of the pulse form provides additional information about ion density and reactive operation mode (metallic, transition, poisoned). Evaluating either data in a combined control algorithm, opens the door for establishing a long-term process control for productions lines. Examples of coating applications for different layer materials and properties will demonstrate the capabilities of the combined control technique for tuning and controlling the plasma properties to secure constant layer properties.

Abstract 10

Emerging power supply technologies facilitate enhancements of magnetron sputtering for demanding complex thin films.

Gerhard Eichenhofer[*1], Gerd Moser[2], Michael Schweiger[2]; Michael Banghard[3] ; Helen Steins[3]

[1] 4A-PLASMA, Holzgerlingen, Germany

[2] J.Schneider Elektrotechnik, Offenburg, Germany;

[3] NMI Natural and Medical Sciences Institute at the University of Tübingen, Germany

*Corresponding author
E-mail: eichenhofer@4a-plasma.eu

For flexible, stretchable and temperature sensitive, functional substrates, obviously coatings need to be more and more sophisticated. This especially is true for micro medical implants into the human body.
There are of course several ways to position the lever for improvements. For power delivery systems, the introduction of asymmetrical bipolar pulsing in the 90's has already been a great step into the right direction. This has been implemented very effective, mainly to discharge the target for DC-pulsed depositioning and/or to prevent and handle arcing. In recent years the reverse positive voltage impulse has been introduced successfully by ambitious organizations for state of the art HiPIMS applications. This even further enhances the film properties, opens new process windows and shows increased deposition rates. Additionally, it enables HiPIMS depositioning for non-metallic, nonconductive substrates which attractively expands the HiPIMS field of applications to new technologies. For applications where neither substrate heating nor BIAS -voltage can be applied, as example for functional micro medical implants, polymers and other sensitive materials, the right reverse voltage impulse shows its true value.
It can add controlled energy already in the film nucleation phase and continuously improves the growing film by ion impacts. Above is not only true for HiPIMS.
If regulated and adjustable voltages, neg. and reversely positive, are implemented into an asymmetrically bipolar DC-pulsing PS unit which is designed as a true current source, most outstanding defect free oxide films have been demonstrated. Micro arcing, even on highly arc susceptive oxynitride coatings, is literally eliminated.
In this work, the history of the positive reverse voltage pulse and its effects will be reviewed. It will be shown (i) how different positive voltages can significantly impact the overall film characteristics for HiPIMS and DC-Pulsing, thus enhancing the performance of the final coating, (ii) and how a purely current regulated DC-pulse power supply technology compares to conventional technologies.

Abstract 11

DSMC simulations of sputtered particle flux and gas rarefaction in DC and HIPIMS magnetron sputtering

P. Zikán[1] , A. Obrusník[1] , J. Hnilica[2] , N. Britun[3] , P. Jelínek[1] , P. Vašina[2]

[1] PlasmaSolve Company, Brno, Czech Republic,[2] Department of Physical Electronics, Masaryk University, Brno, Czech Republic
[3] Chimie des Interactions Plasma-Surface (ChIPS), CIRMAP, Universite de Mons, Belgium

*Corresponding author
E-Mail: zikan@plasmasolve.com

The DSMC method is a well-established numerical method, originally invented for modeling supersonic gas flows.
It was, however, shown that the method can also be useful for various PVD processes [1]. Once correctly set up, these simulations can provide useful insight into sputtered neutral material transport and its interplay with the background gas. The implementation requires support for complex 3D geometries, should this method be relevant to industrial applications. Luckily, there are several open-source implementations of the method available. The most versatile one is probably dsmcFoamPlus solver [2].
This solver supports 2D, 2D-axi and 3D geometries, multi species simulations and powerful probing and measuring techniques. High abstraction and modularity of the code enables implementation of new boundary conditions and collision schemes to make the solver applicable to magnetron sputtering challenges. The presence of the background gas(es) poses a challenge during simulation of transport of the neutral sputtered material through a vacuum chamber. Background gas density is usually much higher than the density of sputtered species.
Therefore, in order to resolve the transport of the latter, one needs very large number of background gas simulator particles. This can be a serious limitation in terms of hardware requirements, especially in 3D geometry. To overcome this limitation, an enhanced collisional scheme for the dsmc method was implemented. Assuming that the chamber is filled with uniform background gas which is not heated

substantially by the sputtered atoms, background gas particles can be treated only virtually by sampling them from Maxwell-Boltzmann distribution only when necessary. This dramatically reduces the CPU and memory requirements of simulations where the metal vapours are highly dilute. On the other hand, during HiPIMS pulse, the density of sputtered material is comparable to the density of the background gas. This allows to simulate the sputtering process self-consistently with the background gas and to capture rarefaction in front of the target and it's temporal evolution. Influence of the background gas density and the collisional scheme (VHS, VSS, M1) will be presented, together with experimental validation. The experimental data were obtained by laser-induced fluorescence measurements of titanium atom and titanium ion density.

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Abstract 12

T3 or T5: Tungsten based conductive coatings for PEMWE bipolar plates

Lucia Mendizabal

IK4-Tekniker, Iñaki Goenaga 5, Eibar, Spain

***Corresponding author**
E-mail: lucia.mendizabal@tekniker.es

Polymer electrolyte membrane water electrolyzers (PEMWE) coupled with renewable energy resources (wind and solar) represent one of the most promising candidates for clean hydrogen production. Bipolar plates (BPP) are crucial components in PEM electrolyzers because they must provide mechanical support to the cell, separate product gases and possess high thermal and electrical conductivity. BPPs are exposed to acidic environment, but also must withstand high overvoltage (up to 2V) in oxygen atmosphere in the anodic side of a PEM eletrolyzer. These factors strongly reduce the material candidates for BPP fabrication. Titanium (Ti) is considered as the state-of-the-art material and stainless steel (SS) as a potential low-cost candidate for its substitution. SS is not corrosion-resistant in PEM electrolyzer environment and must be coated to ensure an adequate performance. Ti tends to form an insulating passive oxide layer that increases interfacial contact resistance (ICR) and diminishes electrolyzer cell efficiency as electrolyser ages WOx coatings grown under different sputtering process parameters on Ti and SS samples by pulsed dc magnetron sputtering were investigated. Microstructure and composition of different WOx films were correlated with the performance of those coatings exhibited during electrochemical testing and interfacial contact resistance (ICR) measurements in PEMWE environment.

Abstract 13

HiPIMS magnetized plasma afterglow diagnostic

M. Ganciu*, B. Butoi, A. Groza, B. Mihalcea

National Institute for Laser, Plasma and Radiation Physics, PO Box MG-36, 077125, Magurele, Bucharest, Romania,

***Corresponding author**
E-mail: mihai.ganciu@inflpr.ro

At an international level, the deposition of thin films, which are resistant to aggressive conditions, by means of plasma deposition techniques is a technological challenge. For deposition of dense and ultra-dense films there exist High-power impulse magnetron sputtering (HIPIMS) which ensure highly ionized pulverised vapours and high molecular dissociation [1]. Many studies were developed on phenomena associated with magnetized-plasma high ionization, which lead to a high energy impulse deposition on the cathode surface (1-3 KW/cm2), with an average deposited energy 1-10 W/cm2 on the cathode [2]. An interesting application was high quality optical depositions [3], and, recently, it was demonstrated that by using a short-pulse HiPIMS technique, one can obtain thin films with superior characteristics, which can resist in extreme conditions, specific to the cosmic space [4]. DLC coatings by HiPIMS technique [5] could also be useful for space applications [6]. The definite advantages of working with ultra-short pulses are associated with obtaining high-qual-

ity deposited films: denser films, with a good adhesion, lower thermal load of the substrate, a better coverage of complex surfaces, avoiding the passage to auto-pulverisation, stable and reproducible reactive processes, relaxation times of the deposited surfaces which are compatible with ion packages deposited in short pulses, etc. [7, 8].
The response of the HiPIMS equipment to intense pulses of the cathodic current is essential for identifying/realizing adequate electric HIPIMS systems. The pre-ionisation turned out to be useful in reducing the building time of the magnetized plasma, which controls the cathodic current for an optimal cathodic voltage. The initial, low-density magnetized plasma can be obtained by DC discharges, pulse succession and RF discharges [6]. For establishing the optimal working conditions it is useful to know the lifetime of the magnetized plasma at the end of the cathodic pulsed current. The table-top system developed by us allows one to measure this time for all working HiPIMS types, both for short and long pulses, being based on the rapid increase of the current on the application of the voltage pulse with very short rise time, to a value which depends on the characteristics of the magnetized plasma [9]. At the same time, the system allows one to define the working parameters for the input system in HiPIMS regime for various applications. A brief analysis of these aspects is done in the paper, the first experiments are described by using fast-regime HiPIMS equipment and an interpretation of the results is given.

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Abstract 14

On three different ways to quantify the degree of ionization in sputtering magnetrons

Daniel Lundin[1] *, Alexandre Butler[1], Nils Brenning[1,2,3], Michael A Raadu[2], Jon Tomas Gudmundsson[2,4], Tiberiu Minea[1],

1 Laboratoire de Physique des Gaz et Plasmas—LPGP, UMR 8578 CNRS, Université Paris-Sud, Université Paris-Saclay, F-91405 Orsay Cedex, France, 2 Department of Space and Plasma Physics, School of Electrical Engineering and Computer Science, KTH Royal Institute of Technology, SE-100 44, Stockholm, Sweden, 3 Plasma and Coatings Physics Division, IFM-Materials Physics, Linköping University, SE-581 83, Linköping, Sweden, 4 Science Institute, University of Iceland, Dunhaga 3, IS-107 Reykjavik, Iceland

***Corresponding author**
E-Mail: daniel.lundin@u-psud.fr*

Quantification and control of the fraction of ionization of the sputtered species are crucial in magnetron sputtering, and in particular in high-power impulse magnetron sputtering (HiPIMS), yet proper definitions of the various concepts of ionization are still lacking. In this contribution, we distinguish between three approaches to describe the degree (or fraction) of ionization: the ionized flux fraction F_flux, the ionized density fraction F_density, and the fraction α of the sputtered metal atoms that become ionized in the plasma (sometimes referred to as probability of ionization).
By studying a reference HiPIMS discharge with a Ti target, we show how to extract absolute values of these three parameters and how they vary with peak discharge current. Using a simple model, we also identify the physical mechanisms that determine F_flux, F_density, and α as well as

how these three concepts of ionization are related. This analysis finally explains why a high ionization probability does not necessarily lead to an equally high ionized flux fraction or ionized density fraction.

Abstract 15

Design of Experiment methods as an effective tool in industrial implementation of HIPIMS technology

Wojciech Gajewski[1], Robert Mroczyński[2], Marek Betiuk[3], Mirosław Puźniak[1,2], Piotr Domanowski[4], Piotr Różański[1], Marcin Żelechowski[1]

1 TRUMPF Huettinger Sp. z o.o., Marecka 47, 05-220 Zielonka, Poland, 2 Institute of Microelectronics and Optoelectronics, Warsaw University of Technology, Koszykowa 75, 00-662 Warsaw, Poland, 3 Department of Advanced Technologies, Institute of Precision Mechanics (IMP), Duchnicka 3, 01-796 Warsaw, Poland, 4 Department of Mechanical Engineering, UTP University of Science and Technology, Prof. Sylwestra Kaliskiego 7, 85-796 Bydgoszcz, Poland

***Corresponding author**
E-mail: wojciech.gajewski@trumpf.com

Due to an extensive investigation performed over last decade the High Power Impulse Magnetron Sputtering (HIPIMS) technology has been now accepted as mature for industrial scale production of coatings with superior microstructure and properties. As the properties of a magnetron sputtered coating depend on a number of parameters including gas pressure, process temperature or average power an implementation of HIPIMS generator into the process recipe extends the parameter list to be considered. Furthermore, the selection of the proper parameter value cannot be made without testing various combinations of thereof. For example, to investigate influence of the combination of three values of pressure, gas ratio, temperature and average power would normally require 3^4 i.e. 81 test runs. Including only basic user-adjustable parameters in modern HIPIMS power supplies such as peak current, pulse length and frequency extends the parameter combination matrix even further, thus, generating an unacceptable financial and time burden for introducing HIPIMS into production. This study reports on application of the Taguchi orthogonal table method for justification of the HIPIMS technology at a typical production line conditions. Optimization of the morphology and optical parameters at a rduced number of experiments using Taguchi approach will be analyzed using two model Ti-based coatings used in variety of industrial branches as decorative, optical or protective coatings: TiN and TiOx.
It will be shown that by using Taguchi method the number of test runs necessary to identify the influence of process (pressure, temperature, gas ratio) and power supply-related parameters (average power, peak current, pulse length and frequency) on coating properties can be dramatically decreased.
Finally, the suitability of the Taguchi method to indicate the best parameter value configuration for optimization of selected coating property will be discussed.

Abstract 16

A novel industrial coating system for the deposition of smooth hard coatings combining HiPIMS V+ and rotatable magnetrons

Herbert Gabriel[1]*, Ivan Fernandez, Jose-Antonio Santiago*, Ambiörn Wennberg****

**PVT Plasma und Vakuum Technik GmbH, Bensheim, Germany*
***Nano4Energy, Madrid, Spain*

***Corresponding author**
E-Mail: h.gabriel@PVTvacuum.de

Multi-layered, nano-structured metal-nitride and carbonitride coatings are very well established in the cutting tool industry as well as in other industries. For years most of such coatings have been deposited by arc evaporation despite the badmouthed “droplets”, since arc evaporation is an extremely economic process with significant advantages such as high intrinsic ionization which is particularly beneficial during metal etching. Magnetron sputtering with its low ionization and its deficiencies in adhesion and productivity significantly improved with the development of HiPIMS. An even more significant improvement is the HiPIMS V+ process where adding positive reverse pulses creates enhanced ion assistance and incorporation to the growing film, thus also increasing the deposition rate. On the other hand, rotatable magnetrons are well known to provide better material usage, longer operation and higher operation power levels. The novel industrial system introduced on this paper shows the unique combination of HiPIMS V+ with rotatable magnetrons in a batch coater system, thus enhancing system productivity. This process is applied in a multi-cathode magnetron sputtering system using 4 pieces of 1 m long rotatable cathodes equipped with a strong unbalanced magnetic design allowing high ion-to-neutral ratios to the substrate. The system can be configured to operate in unipolar HiPIMS, Dual Bipolar HiPIMS or DC-Pulsed. Besides a description of the newly designed coating system, nitride and carbonitride nano-structured multilayered coating structures based on Ti, AlTi, AlCr and TiSi deposited in such system are shown and characterized, conceing their microstructure, adhesion, microhardness and composition.

Abstract 17

HIPIMS with positive voltage reversal: a method for influencing the coating properties on insulating substrates.

Ambiörn Wennberg and Iván Fernández

Nano4Energy SL and hip-V Joint Venture, C/ Jose Gutierrez Abascal 2 ES-28006 Madrid, Spain.

***Corresponding author**
E-mail: ambiorn@nano4energy.eu

Current engineering and material advances are shifting manufacturing in many areas from solid bulk materials to flexible lightweight materials. Although these materials, such as polymers, are lightweight, flexible and tough, there are challenges to engineering coatings on such substrates as they insulating and not able to withstand high temperatures. This gives rise to the challenge of how to deposit high quality thin film coatings on such substrates. High Power Impulse Magnetron Sputtering (HIPIMS) has shown many advantages over conventional sputtering which is commonly used to deposit metals, metal nitrides and metal oxides on polymer web. With HIPIMS, a fraction of the target material will be ionized while the ion energy distribution function will shift to energies about 10 times greater than those for DC discharges. However, this increase in ionization and energy will give only modest changes on an unbiased substrate. With the addition of a positive voltage reversal pulse adjacent to the negative HIPIMS sputtering pulse, these ions can be accelerated towards the substrate providing energy for film nucleation and densification. In this study, improvements in film properties in optical coatings such as film density, index of refraction extinction coefficient and barrier properties are presented for several coatings such as TiOx, TiN, ZnSnOx deposited in different Industrial scale production machines.

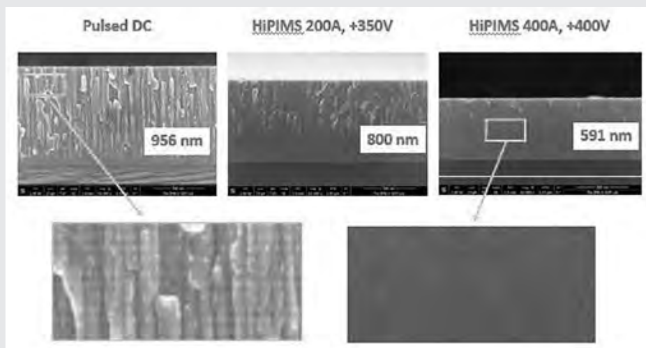


Figure. SEM images of TiOx layers deposited onto Silicon at RT by both Dc-Pulsed and HiPIMS with voltage reversal.

Abstract 18

Study of Molybdenum Plasma by HIPIMS

Daniel A. L. Loch*, Arutiun P. Ehiasarian

HIPIMS Technology Centre, Sheffield Hallam University, Sheffield, UK

***Corresponding author**
E-Mail: d.loch@shu.ac.uk*

Molybdenum is a good candidate as back contact for CIGS cells, due to the appropriate bandgap, low resistivity and good reflectivity of Mo thin films. Compared to conventional PVD processes, HIPIMS offers more control over the thin film properties due to the metal ion dominated plasma.

The behaviour of gas species and their influence on the coating properties is not well understood. A complete understanding of the HIPIMS plasma can support the tailoring of better-performing back contacts. In this study we report the findings of the plasma being analysed by current-voltage (I-V) waveform evaluation, time-resolved plasma sampling energy-mass spectroscopy and optical emission spectroscopy. A voltage-pulse time matrix was devised varying the voltage from 800 - 1500V and the pulse time was increased from 60 - 1000µs in 5 steps. Processes were operated at 0.22Pa and 0.44Pa. The increase and drop of the current within the pulse re-sulted in a change in Ar2+/Ar1+ flux. Mass spectroscopy measurements were taken half-way through the pulse. The Ar1+ IEDF exhibits a low energy peak representing thermalised ions at 1.5eV. With increasing pulse time, the peak intensity increases by 2 orders of magnitude and a higher energy tail up to 15eV develops. Ar2+ IEDF displays a broad peak between 5-15 eV, with a higher energy tail to 25eV. With increasing pulse time the tail disappears and the peak reduces by an order of magnitude (Fig.2).

The ratio of the Ar2+/Ar1+ intensity is larger for shorter pulses and higher currents, and reduces with increasing pulse time where the current reduces to a plateau. This indicates that due to gas rarefaction in the beginning of the pulse, a significant fraction of double-charged gas ions is created that can sustain high currents and higher sputter rates on account of the higher charge and energy of bombardment. To get further insight into the excited atom evolution with pulse time and voltage, we will also be discussing the OES results.The presented results reveal new details of the gas rarefaction process and illustrate the control potential for HIPIMS plasma in support of the development of better performing back contacts.

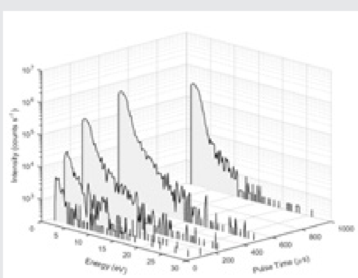


Figure 1:
IEDF's of Ar1+ at 800V and 0.22Pa for pulse widths of 60-1000µs.

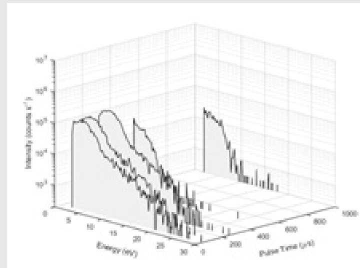


Figure 2:
IEDF's of Ar2+ at 800V and 0.22Pa for pulse widths of 60-1000µs.

Abstract 19

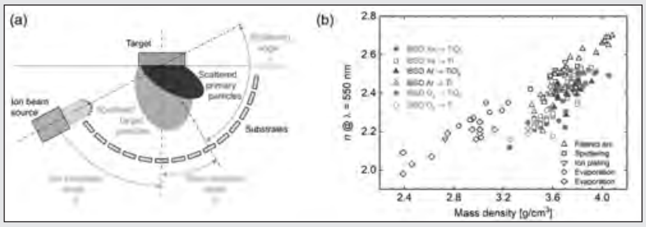
Options to Tailor Thin Film Properties by Ion Beam Sputter Deposition

Carsten Bundesmann*, Thomas Amelal, René Feder, Daniel Spemann

Leibniz-Institute of Surface Engineering (IOM), Permoserstr. 15, 04318 Leipzig, Germany

***Corresponding author**
E-Mail: carsten.bundesmann@iom-leipzig.de

There is an increasing demand for thin films with optimized properties, for instance, for optical, electronic, sensor or energy conversion applications. Likewise, the requirements concerning quality and the variety of applications is steadily growing. This requires the use and control of adequate deposition techniques. Ion beam sputter deposition (IBSD) is a PVD technique that is capable to fulfil the technological challenges, because it offers the unique opportunity for tailoring the properties of film-forming particles and, hence, thin film properties over a wide range /1/. This is related to the fact that the generation and acceleration of the primary particles (ion source), the generation of the film-forming particles (target) and film growth (substrate) are spatially separated. Thus, by changing ion beam parameters (ion species, ion energy) and geometrical parameters (ion incidence angle), the angular and the energy distribution of the sputtered target particles and scattered primary particles are modified.



(a) Sketch of experimental IBSD setup. (b) Index of refraction at λ = 550 nm versus mass density of TiO2 thin films grown by IBSD and, for comparison, various other PVD techniques.

Systematic investigations of the correlation of process parameters, the properties of the sputtered and scattered particles, and thin film properties were performed. Several materials, including TiO2 and SiO2 (dielectrics) and Ag (metal), were focus of the studies. Though the materials are very different, the fundamental systematics were found to be the same. The main results can be summarized as follows. (i) Sputtered and scattered particles can gain a maximum energy of several 100 eV, in case of scattered particles even higher than 1000 eV. (ii) The most important process parameters are the scattering geometry and the primary ion species, or, to be more precise, the mass ratio of primary and target particles. Ion energy and ion incidence angle have only a small impact. (iii) High-energy scattered primary particles constitute an „intrinsic ion-assist" and have a strong influence on thin film properties. Merely by choosing the process parameters appropriately, the ion assist can be switched on/off and adapted to the technological needs. The results are illustrated by selected examples. The influence of the process parameters on the energy distribution of the film-forming particles and on the thin film properties, such as, structural properties, composition, surface roughness, mass density, electrical or optical properties is shown.

/1/ C. Bundesmann, H. Neumann: Tutorial: The Systematics of Ion Beam Sputtering for Deposition of Thin Films with Tailored Properties; J. Appl. Phys., 124 (2018) 231102, DOI: 10.1063/1.5054046. Germany

Abstract 20

Roll-to-Roll Gas Flow Sputter Deposited Copper

Chun-Ming Chen[1], Sz-Ying Chen[1], Ying-Hung Chen[2], Ping-Yen Hsieh[2], Tsung-Han Chen[2], Kuo-Bing Cheng[3], Ju-Liang He[1,2]*

[1]Department of Materials Science and Engineering, Feng Chia University, No. 100, Wenhwa Rd., Seatwen District, Taichung City 40724, Taiwan.[2]Institute of Plasma, Feng Chia University, No. 100, Wenhwa Rd., Seatwen District, Taichung City 40724, Taiwan. [3]Department of Fiber and Composite Materials, Feng Chia University, No. 100, Wenhwa Rd., Seatwen District, Taichung City 40724, Taiwan.

***Corresponding author**
E-Mail: jlhe@o365.fcu.edu.tw

Echoing the strong demand in flexible copper clad laminate (FCCL) for circuitry utilization in those mobile devices, PVD, sputtering in particular, found its way to fulfill somehow in the two-layer FCCL with advantages in fine pitch etching and dimensional accuracy. However, the low deposition rate and relatively poor film adhesion of this approach is one major technical concern for its usage, let alone the hazardous wet reagents used for the post electroplating. An attempt in this work is to employ a roll-to-roll (R2R) gas flow sputtering (GFS) for obtaining two-layer FCCL. A 25 cm GFS source was mounted onto a coating chamber door and a R2R winding-rewinding system was installed to take advantage of continuous production. Pure copper was sputtered on polyimide (PI) substrate. The results show that when operated at a dc power level of 8 kW with an argon flow rate of 12 slm, the R2R-GFS process can reach a film thickness of 200 nm at a web travelling linear speed of 50 cm/min. The electrical surface resistance of the copper film on PI can ultimately reach a level of 10-3 Ω/□ and can strongly be affected by substrate bias voltage.
It was found that the film structure can be varied greatly by biasing the substrate. The copper film adhesion on PI substrate tested by a Scotch-tape test can be ranked to achieve the highest level of 5B.

Keyword: flexible copper clad laminate (FCCL), gas flow sputtering (GFS), roll-to-roll (R2R)

Abstract 21

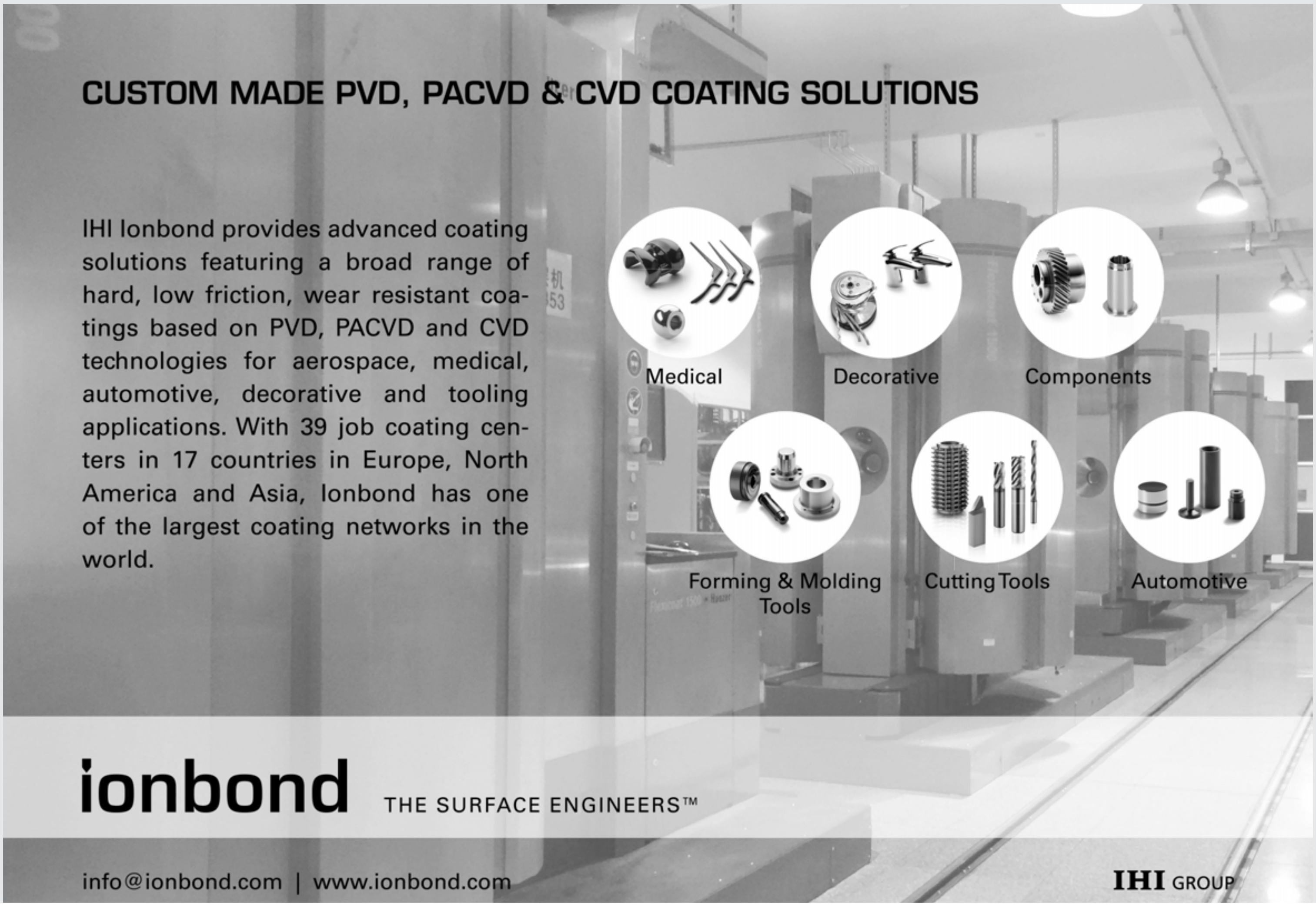
Plasma Chemistry, Crytal Growth and Mechanical Properties of CrAlYN / CrN Nanoscale Multilayer Coatings Deposited by High Power Impulse Magnetron Sputtering

A.P. Ehiasarian, A. Sugumaran, P.EH. Hovsepian

National HIPIMS Technology Centre, Sheffield Hallam University, Howard St., Sheffield, S1 1WB, UK

***Corresponding author**
E-Mail: a.ehiasarian@shu.ac.uk

Nanoscale multilayer coatings based on CrAlYN / CrN find applications in manufacturing, automotive components, power generation turbines and petrochemical industry.
To perform well in these different environments, the coating microstructure must be tailored via the deposition process.
In High Power Impulse Magnetron Sputtering (HIPIMS), which provides a high degree of ionisation of the metal flux and activation of the reactive gas, the relation between process parameters, microstructure and coating properties is not well understood. We report on the effect of unbalancing magnetic field on species-dependent transport of metal and gas species to the substrate and its influence on film growth, texture formation and mechanical performance of nanoscale multilayer CrAlYN/CrN films. Experiments were carried out in an industrial-sized coater with four cathodes arranged in a closed magnetic field configuration, two of which were operated in HIPIMS mode and two in conventional sputtering. In a balanced configuration, the magnetic null height was hm = 12 cm and the volume of plasma near the target was the greatest, extending beyond the cathode presheath and resulting in a high metal-to-gas ion ratio (JMe+ / JG+) observed by optical emission spectroscopy. The transport to the substrate, as measured by the substrate ion current, was the lowest due to the absence of magnetic field lines connecting to the substrate. The 4-micrometre-thick films exhibited competitive growth and a strong [111] texture evidenced by XRD due to the relatively low flux of dissociated nitrogen to the surface. SEM observations showed that the [111] texture resulted in dome-shaped column tops and clearly defined column boundaries where vacuum impurities were segregated.
As the magnetic field grew more unbalanced, the confinement volume steadily decreased whilst transport to the substrate was enhanced, resulting in both JMe+ / JG+ and ion flux to the substrate (Ji) reaching their maximum values. Even weakly unbalanced fields with hm = 10 cm provided sufficient flux of activated species to cause the grains to switch rapidly to [220] and then to [200] texture and allowing them to absorb impurities interstitially. This resulted in the elimination of dome-shaped morphology, drastic reduction in roughness, parallel column boundaries and increase in grain size. Highly unbalanced fields (hm = 4 cm) constricted the height of the confinement volume below the thickness of the ionising presheath, reducing the ionisation of metal and dissociation of nitrogen as evidenced by the significant reduction in JMe+ / JG+.
There was only a slight loss in overall current, Ji, as it was compensated by ionisation of the gas outside the confinement zone. The loss of dissociation switched the texture back to a strong [111]. Grain sizes were significantly larger than for the balanced configuration due to a higher Ji.
The dry sliding friction coefficient against Al2O3 reached as low as 0.61 for the balanced configuration. The hardness and wear rates are discussed.


CUSTOM MADE PVD, PACVD & CVD COATING SOLUTIONS
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POSTER PRESENTATION

Abstract 22

Spatial and temporal measurements of plasma parameters in a bipolar HiPIMS discharge

F. Walk*[1], R. Valizadeh[2], J.W. Bradley[1]

[1] *Department of Electrical Engineering and Electronics, University of Liverpool, Brownlow Hill, Liverpool, L69 3GJ, United Kingdom,* [2] *STFC Daresbury, Warrington, Cheshire WA4 4AD, United Kingdom*

***Corresponding author**
E-mail: Felix.Walk@liverpool.ac.uk

Langmuir probe measurements were carried out in a 3 inch magnetron system with a niobium target operating in argon to determine how positive polarity pulses in the HiPIMS voltage waveform can change the dynamics of the plasma. The positive voltage pulses were introduced in the early afterglow with heights Upos between 10 and 100V and a variation of durations between 10 and 300 µs.
Operating argon pressures were varied from 5 to 24mTorr. The plasma parameters were determined with a time-resolution of 2:5 µs during all stages of the pulse cycle (100 Hz duration) along two lines-of-sight in the discharge, namely in the axial diretion above the centre line and above the racetrack.
Probe measurements of the evolving ion density nidistribution (determined using a Laframboise analysis) show clearly the eect of the positive polarity pulse, indicating enhanced ion repulsion from the cathode. In the on-time of the HiPIMS pulse, maximum values of ni up to 1019 m^{-3} were measured for average powers of 200W, with ni falling by an order of magnitude over 100 µs into the positive voltage reversal phase. Plasma potential Vp measurements reveal a monotonic rise after initiation of the positive voltage reversal to values close to Upos with the time for transition determined by the operating pressure and distance from the target. Interpretation of the results in terms of fundamental magnetron plasma processes will be discussed as well as implications of biplar pulsing for enhanced thin-film growth.

Abstract 23

Overstoichiometric $TMN_{x>1}$ transition metal nitrides

Zuzana Čiperová*, Jindřich Musil, Šimon Kos, Martin Jaroš

Department of Physics and NTIS - European Centre of Excellence, University of West Bohemia, Univerzitní 8, 306 14 Plzeň, Czech Republic

***Corresponding author**
E-mail: ciperovz@kfy.zcu.cz

The work reports on formation of strongly overstochiometric ZrNx>1 and Ti(Al,V)Nx>1 coatings by reactive magnetron sputtering [1]. Problems in the formation of overstoichiometric coatings and possible ways to form strongly overstochiometric TMNx>1 nitride coatings up to TMNx=2 dinitride coatings are discussed; here TM are the transition metals such as Ti, Zr, Mo, Ta, Nb, W, etc. The coating stoichiometry x = N/TM strongly influences its electrical and mechanical properties. The creation and properties of reactively sputtered ZrNx coatings are presented in detail. It was found that (1) the electrical resistivity of the ZrNx coating varies with increasing x from well electrically conducting films with x ≤ 1 through the semi-conducting films with x ranging from 1 to ≤ 1.26 to non-conductive with x ≥ 1.3, showing that the stoichiometry x is a strong parameter which enables to control an electric conductivity of the coating in a wide range; (2) the electrically conductive coatings with x ≤ 1 are harder than the semiconducting and electrically insulating coatings; and (3) the ZrN2 dinitride film cannot be created due to the formation of Zr3N4 phase whose formation enthalpy is greater than that of a ZrN2 phase. Also, it is shown that a main problem in the formation of strongly overstoichiometric TMNx>1 and dinitride TMN2 coatings is a strong increase of ionization of the nitrogen sputtering gas to achieve a necessary high ratio N/TM > 1.

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Abstract 24

On the effect of stationary magnetic field on spatial distribution of deposition rate and ionized flux fraction in the HiPIMS discharge

H. Hajihoseini[1,2] and J. T. Gudmundsson[1,2]

Science Institute, University of Iceland, Dunhaga 3, IS-107 Reykjavik, Iceland 2 Department of Space and Plasma Physics, School of Electrical Engineering, KTH-Royal Institute of Technology, SE-100 44, Stockholm, Sweden

***Corresponding author**
E-mail: tumi@hi.is

We have explored the effect of magnetic (B) field strength and geometry on the deposition rate and ionized flux fraction (IFF) of HiPIMS deposited titanium. We applied a 4 inch. magnetron capable of a varying the absolute B-field strength as well as the geometry of the B-field (degree of balancing) by adjusting two micrometer screws located externally. The magnetron gun as well as one probe holder are mounted on movable bellows controlled with millimeter precision. This makes it possible to perform radial as well as axial scans with high precision. The HiPIMS discharge was run in two operating modes. The first one we refer to as 'fixed voltage mode' where the cathode voltage is kept fixed at 500 V while the pulse frequency is varied to achieve the desired time average power (300 W). The second mode we refer to as 'fixed current mode' is carried out by adjusting the cathode voltage while keeping the peak current at 40 A as well as varying the frequency to archive the same average power. Our results show that the dcMS deposition rate is barely sensitive to the B-field variations while the deposition rate during HiPIMS operated in fixed voltage mode depends on the B-field and changes from 30% to 90% of the dcMS rate. However, when operating in the HiPIMS fixed current mode the deposition rate is almost independent of the B-field strength while it is sensitive to the degree of balancing. In terms of IFF, in fixed voltage mode, the higher the deposition rate, the lower the IFF. Although in the fixed current mode, at the same deposition rate, the weaker magnetic strength results in a higher IFF. Thickness uniformity measurements illustrate that the dcMS deposition rate is hardly sensitive to the B-field while both HiPIMS operating modes are highly sensitive to the B-field. The HiPIMS deposition rate uniformity can be 10 % lower up to 10 % higher than the dcMS rate depending on the B-field.

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Abstract 25

Time-resolved Langmuir probe diagnostics carried out during the positive voltage pulses in bipolar HiPIMS discharges

A.D. Pajdarová[1*], T. Kozák[1], J. Čapek[1], P. Mareš[2], M. Čada[3] and Z. Hubička[3]

[1]Department of Physics and NTIS – European Centre of Excellence, University of West Bohemia, Univerzitní 8, 306 14 Plzeň, Czech Republic ; [2]HVM Plasma, s.r.o., Na Hutmance 2, 15800 Prague 5, Czech Republic; [3]Institute of Physics, Academy of Sciences of the Czech Republic, Na Slovance 2, 182 21 Prague 8, Czech Republic

***Corresponding author**
E-mail: adp@kfy.zcu.cz

Nowadays, bipolar HiPIMS discharges have attracted HiPIMS community attention. The application of the positive voltage pulse after the main high-power negative voltage pulse allows not only the charge neutralization on the target during the deposition of dielectric films (to avoid arcing), but it also increases the energy of ions flowing onto the substrate, which may induce an improvement in the film properties. Here, we mainly report on the results of time-resolved Langmuir probe diagnostics carried out during the positive voltage pulses in bipolar HiPIMS discharge. A self-made bipolar HiPIMS source

providing almost rectangular positive voltage pulses (with a controllable amplitude, delay after the main high-power negative voltage pulse, and positive pulse duration) was connected to an unbalanced circular magnetron (Ti target, the diameter of 100mm). During the experiments, the parameters of the negative high-power voltage pulse were kept constant (the pulse duration of 100µs, the pulse-averaged target power density of 1kWcm-2, and the repetition frequency of 100Hz). The probe tip (cylindrical, the length of 10mm and diameter of 0.15mm) was located at the discharge axis in the distance of 100mm (the typical distance of the substrate in our experiments) from the target, and it was parallel with the target surface. To protect the probe tip against its damage caused by high currents during the negative high-power voltage pulse at the high probe potentials (up to 200V to the ground) an electrically controllable fast switch allowing disconnection of the probe tip from the probe voltage source during the negative high-power voltage pulse was employed.
The probe current-voltage characteristics were reconstructed from the waveforms of probe currents recorded during the positive voltage pulses for a gradually increasing probe potential. This procedure allows us to reach a sub-µs time resolution. From the probe current-voltage characteristics the time evolution of plasma parameters (the plasma and floating potentials, the density of electrons and their temperature) were derived.
We examined the influence of an alternation of the positive voltage pulse settings (the positive voltage amplitude, the delay between the negative high-power voltage pulse and positive voltage pulse, and the duration of positive voltage pulse) on those plasma parameters. The acquired results together with the outcomes of time- and energy-resolved mass spectroscopy and time-resolved optical emission spectroscopy imaging allowed us to reveal plasma processes taking place during the positive voltage pulses and assessed the effects of these plasma processes on the deposition of films.

Abstract 26

Photocatalytic Ta-O-N films prepared by reactive HiPIMS

Š. Batková[1], J. Čapek[1], S. Haviar[1], J. Houška[1], R. Čerstvý[1], M. Krbal[2], T. Duchoň[3]

1Department of Physics and NTIS - European Centre of Excellence, University of West Bohemia, Univerzitní 8, 306 14, Plzeň, Czech Republic; 2CEMNAT, University of Pardubice, nám. Čs. legií 565, 530 02, Pardubice, Czech Republic 3Department of Surface and Plasma Science, Charles University, V Holešovičkách 2, 180 00, Praha, Czech Republic

***Corresponding author**
E-Mail: sbatkova@kfy.zcu.cz

The TaON and Ta3N5 materials are both promising candidates for application as a visible-light-driven photo-catalyst splitting water into H2 and O2. In order to work as a water splitting photocatalyst, the material must satisfy certain conditions: (i) band gap of proper width (preferably corresponding to visible light absorption) and (ii) suitable alignment of the band gap with respect to the water splitting redox potentials. The subsequent transport of the charge carriers through the material (particularly across the films thickness) plays an important role in the effectivity of the process. In this work we first demonstrate that using reactive high-power impulse magnetron sputtering (HiPIMS) as the deposition technique followed by post-annealing of the amorphous as-deposited film at 900°C in a vacuum furnace allows us to prepare a polycrystalline film exhibiting a pure TaON or Ta3N5 phase. Such films satisfy the above mentioned conditions for a water splitting photocatalyst. However, as it is desirable to prepare these phases in situ, we investigate the possibilities of using different substrates and/or substrate heating during deposition. We present photoelectrochemical measurements of the films and report on films modified by metallic nanoclusters, designed to suppress the recombination rate of the photo-generated electron-hole pairs.

Abstract 27

YmOn and YTTRIUM Doped ZnO thin films and multilayers

Halil Arslan, Andris Azens, Martins Zubkins, Juris Purans

Thin Films Laboratory, Institute of Solid State Physics, University of Latvia

***Corresponding author**
E-mail: halil@cfi.lu.lv

Although doped ZnO thin films are promising n-type TCO materials, obtaining p-type ZnO thin films is an important milestone in the development of transparent electronics. Recently we have developed p-type ZnO-IrO2 thin films [1,2] and compared with the published theoretical models. Unfortunately, iridium is a very expensive material. Therefore, alternative doping element will be greatly demanded. In the scope of this research we are aiming to develop innovative ZnO:Y (YZO) and multilayers ZnO/YO/ZnO (DMD and DM) onto Si (100), Ti and Glass substrates by Magnetron sputtering under extreme conditions. Only recently [3], it was discovered new phase solid phase rock salt structure yttrium monoxide, YO, with unusual valence of Y2+ (4d1) was synthesized in a form of epitaxial thin film by means of pulsed laser deposition means. In contrast with Y2O3, YO was dark-brown colored and narrow gap semiconductor. The tunable electrical conductivity ranging from 10-1 to 103 Ω-1cm-1 was attributed to the presence of oxygen vacancies serving as donor of electron. To understand the local electronic and atomic structures in conjunction with their physical-chemical properties advanced in-lab techniques such as XRD, Raman and FT-IR is performed.

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Abstract 28

Cryogenic deposition of thin films by reactive magnetron sputtering

M. Zubkins*, J. Gabrusenoks, G. Cikvaidze, L. Bikse, J. Purans

Institute of Solid State Physics, Kengaraga 8, Riga, Latvia

***Corresponding author**
E-Mail: zubkins@cfi.lu.lv

The microstructure, as well as the atomic structure, of thin films deposited by magnetron sputtering strongly depends on the growth temperature and the particle bombardment. The mobility of the adatoms increases with the temperature and the crystallites tend to grow larger with the thermodynamically stable atomic structure. However, from a practical point of view, it would be important to develop processes that make it possible to produce structures that cannot be obtained at room or higher temperatures.
It is expected that by decreasing the deposition temperature down to cryogenic temperature, it would be possible to obtain amorphous or metastable structures with new material properties. Zinc oxide (ZnO) exhibits a strong trend to form crystalline phases, mainly the wurtzite structure. The zincblende and rocksalt structures are also thermodynamically stable, but hard to synthesize. From theoretical calculations, 3-, 4- and 5-coordinated ions are present in the amorphous zinc oxide (a-ZnO). Therefore, other crystal motifs consisting of different polymorphs, such as h-BN, bct, sodalite, cubane, should be considered. To synthesize a-ZnO films, the deposition temperature should be decreased, or impurities introduced.
The thin films on glass, Ti and Si substrates were deposited by reactive DC magnetron sputtering from a Zn target in an Ar+O2 atmosphere. The temperature of a substrate holder was varied in the range from 40 to -196 OC. The films have been studied by XRD, SEM techniques, as well as Raman, FTIR and UV-Vis spectroscopies.
The structure of the films changes from a polycrystalline to X-ray amorphous phase when the deposition temperature is decreased. The vibration modes of Zn peroxide (ZnO2) crystal lattice have been observed in the vibration spectra

for the samples deposited at approximately -40 OC.
The vibration bands are wide, indicating an amorphous or nanocrystalline structure. Additionally, O-H stretching and Zn-O-H bending vibrations have been detected when the deposition temperature is -40 OC or lower. The intensity of O22-, O2- un O2 bands in the Raman spectra increases if the deposition temperature is decreased. The obtained results indicate the possible formation of zinc peroxide (ZnO2) phase in the films.

ABSTRACT 29

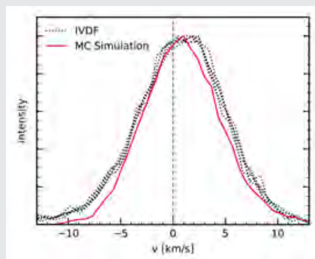
Movement of sputtered particles in the target region of HiPIMS

JULIAN HELD, WOLFGANG BREILMANN, ACHIM VON KEUDELL, VOLKER SCHULZ-VON DER GATHEN

EXPERIMENTAL PHYSICS II, RUHR UNIVERSITY BOCHUM, UNIVERSITÄTSSTRASSE 150, 44801 BOCHUM, GERMANY

***CORRESPONDING AUTHOR**
E-MAIL: JULIAN.HELD@RUB.DE

High power impulse magnetron sputtering (HiPIMS) has been observed to create better coatings than traditional direct current magnetron sputtering but at a reduced deposition rate [2,3]. This has been attributed to the return of ions to the target surface, which in turn is explained by strong electric fields present in the magnetic field trap [4]. Results from modeling indicate, that around 90 % of all ions return to the target surface [5].
Understanding and controlling this “return effect” is the key to improve the deposition rate of HiPIMS. In this work, the movement of titanium ions is investigated. The velocity distribution function (VDF) is obtained using high-resolution optical emission spectroscopy [6]. Doppler broadening of optical emission lines is evaluated to obtain the VDF. The determined VDF is compared to a simple kinetic Monte-Carlo model which is used to simulate the ion movement under the influence of electric fields and Coulomb collisions with other ions.
Good agreement can be reached between measurement and simulation (Fig. 1). We find, that for high target power densities above 1 kW/cm² the influence of the working gas is very weak and the measurement is best reproduced by only considering collisions of titanium ions with other titanium ions. In the direction parallel to the target surface, Coulomb collisions quickly drive the VDF towards a Maxwellian distribution with a high temperature of about 9 eV (Fig 2.). In the direction perpendicular to the target surface, the ion movement is dominated by the electric field (Fig. 1).
A return coefficient of about 94 % is obtained from the model. This value is in line with previous studies [5]. The influence of electric fields and Coulomb collisions on the ion movement is evaluated. Scaling laws for the ion return are discussed and possible ways to optimized the ion flux towards the substrate are compared.
This work has been supported by the German Science Foundation (DFG) within the frame of the collaborative research center SFB-TR 87.



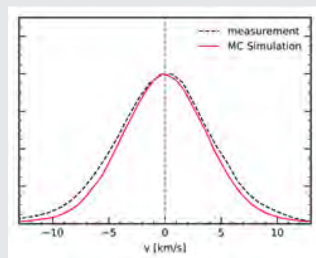


Fig.1 Measured velocity distribution function of titanium ions (IVDF) and the VDF obtained from the model in the direction perpendicular to the target surface.
Fig.2 Measured velocity distribution function of titanium ions (IVDF) and the VDF obtained from the model in the direction parallel to the target surface.

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Abstract 30

Growth of nanostructured ω-Phase Titanium films deposited by biased HiPIMS

D. Dellasega[1], F. Mirani[1], D. Vavassori[1], C. Conti[2], M. Beghi[1], M. Passoni[1]

[1]Department of Energy, Politecnico di Milano, Milan, Italy
[2]ICVBC, National Research Council, Milan, Italy

***Corresponding author**
E-mail: david.dellasega@polimi.it

In the present work the production of titanium (Ti) coatings, deposited by HiPIMS, that exhibit a non-equilibrium hexagonal (ω) crystallographic phase and orientation is presented.
Recent studies have shown that High Power Impulse Magnetron Sputtering (HiPIMS) systems, thanks to the presence of a relevant ionic fraction in the impinging species have great potential for tailoring the properties of the growing film at the nanoscale level. Properly tuning the process parameters it is possible to induce the growth of compact nano-grained films, to influence crystalline growth direction and stress state [1,2]. More specifically, it is reported that the energy of the impinging species, controlled by a proper bias, influences crystallinity, growth direction and in some cases, it may also induce the nucleation of new phases such as tetragonal phase in Ta films [3].
Over many decades, Ti films have been extensively studied and exploited in various fields such as in aerospace, production of medical implants, microelectronics and as adhesion layer for TI oxides and nitrides. Their wide use is related to their exceptional material properties such as diffusion barrier properties, high mechanical strength, excellent chemical and thermal stability and good corrosion resistance. Despite the huge interest on this topic there are few works related to Ti metallic films deposited by HiPIMS [2,4]. In the present work we report the deposition of Ti films at various bias voltages. The energy of the impinging species influences the crystallographic oriented growth of α-Ti (hcp phase), assessed by XRD, as well as residual stresses. At the same time the nucleation of a simple hexagonal phase ω-Ti, is promoted. Such crystalline system is usually obtained out of equilibrium conditions by high pressure torsion at values higher than 5 GPa [5]. The appearance of the ω-Ti phase is related to the presence of elongated grains 100s nm long, detected by SEM, whose features are also related to the deposition time. The elastic properties of the deposited films have been characterized by Brillouin spectroscopy to investigate to role of ω-Ti phase respect to Young modulus.
The obtained Ti films are used to produce compact micrometer and sub-micrometer thick coatings to be used also as free-standing layer. This layers with the addition of an ultra-low-density carbon foam deposited by Pulsed Laser Deposition will be the target materials used in laser driven ion acceleration experiments [6].

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Abstract 31

Effect of magnetron sputtering deposition parameters onto electrical and optical properties of HfOxNy thin films

Mirosław Puźniak[1,2], Robert Mroczyński[1], Wojciech Gajewski[2], Piotr Różański[2], Marcin Żelechowski[2]

1 Institute of Microelectronics and Optoelectronics, Warsaw University of Technology, Koszykowa 75, 00-662 Warsaw, Poland
2 TRUMPF Huettinger, Marecka 47, 05-220 Zielonka, Poland

***Corresponding author**
E-Mail: Miroslaw.Puzniak@TRUMPF.com

High Power Impulse Magnetron Sputtering (HIPIMS technology has been developed over the last twenty years and fulfilled industrial requirements of the hard and decorative coating. The properties of coatings are achieved due to unique control of voltage and current peak shape. It opens the possibility to introduce HIPIMS technology in other commercial applications.

The current development of semiconductor technology requires the design and manufacture of Ultra-Large Scale Integration (ULSI) devices with lower power consumption. It is especially evident in the case of dielectric materials for the gate-stack of MOS/MIS structures, which are widely employed in semiconductor devices [1]. Since 2007, when new dielectric materials characterized by high permittivity values were introduced commercially for the first time, the number of applications of hafnium oxides (HfOx) layers is constantly increasing. There are several possible applications of high-k materials in semiconductor structures and devices, and there are still many important aspects yet to be understood for hafnium-based materials technology and processing [2].

The aim of this study is to analyze and compare the chemical compositions, electrical and optical parameters of Metal-Insulator-Semiconductor (MIS) capacitors with hafnium oxynitride as gate dielectric material. The HfOxNy thin films were deposited by means of reactive sputtering using Pulsed-DC and HIPIMS power supplies. The experimental runs were designed according to Taguchi's orthogonal tables approach [3]. The influence of plasma power delivery on the electrical parameters were investigated by means of capacitance-voltage (C-V) and current-voltage (I-V) measurements. The changes in optical parameters and chemical composition were examined by means of spectroscopic ellipsometry, transmittance and reflectance measurements. Moreover, the influence of Post-Metallization-Annealing (PMA) procedure onto electro-physical properties of fabricated MIS structures, was determined.

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Abstract 32

On ionization fraction of sputtered species

K. Bernátová*, M. Fekete, P. Klein, J. Hnilica, P. Vašina

Department of Physical Electronics, Faculty of Science, Masaryk University, Brno, Czech Republic

***Corresponding author**
E-mail: kbernatova@mail.muni.cz

High Power Impulse Magnetron Sputtering (HiPIMS) is a promising physical vapor deposition technique frequently utilized for the deposition of coatings with enhanced properties. For optimization of the deposition process, the plasma diagnostics is crucial. In this study, Optical Emission Spectroscopy (OES) and Quartz Crystal Microbalance (QCM) is utilized for discharge analysis ranging from standard Direct Current Magnetron Sputtering up to the intensive HiPIMS (duty cycle less than 1%). The QCM system is equipped with a gridless biasable sensor with magnetic electron filter, which allows measuring separately the flux of atoms and flux of the ionized particles impinging the substrate. Effective Branching Fractions (EBF) method is utilized to determine the absolute ground state titanium atom and ion number densities from the self-absorption in plasma measured by OES. The systematic study is provided in three different distances from the target – in the magnetized plasma region, in a region between magnetized plasma and substrate and in the substrate level, all measured in the range of working pressures, at the constant mean power and pulse duration. For intensive HiPIMS at the lowest measured working pressure in the substrate level, the ionized density fraction of sputtered species reaches 70%. At the same conditions, ionized density fraction of sputtered species in the magnetized plasma region reaches 90%. Generally, it was observed that increasing the duty cycle, the peak current density decreases, leading to a decrease of ionized density fraction of sputtered species. Additionally, it was observed that with increasing the working pressure, in the substrate level ionized density fraction of sputtered species slightly decreases for all pulses while for intensive HiPIMS in both magnetized plasma region and transition region remains constant. The ionized metal flux fraction obtained

from QCM measurements are correlated with the ionized density fraction of sputtered species in the substrate level. Both the ionized flux as well as the ionized species density evolutions with the process parameters provide valuable insight into the sputtering process in HiPIMS mode.

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Abstract 33

Spatial distribution of the plasma potential for different magnetron magnetic configurations in HiPIMS with positive pulses.

Ivan Fernandez-Martinez[1], Victor Bellido[2]

[1] Nano4Energy SL and hip-V Joint Venture, C/ Jose Gutierrez Abascal 2 ES-28006 Madrid, Spain., [2] Gencoa Ltd. 4 De Havilland Drive Estuary Commerce Park Liverpool L24 8RN United Kingdom

***Corresponding author**
E-mail: ivan.fernandez@nano4energy.eu

Recently, it has been demonstrated for highly ionized discharges that the application of a positive voltage reversal pulse adjacent to the negative sputtering pulse gives rise to the generation of high fluxes of energetic ions. This effect allowed unprecedented benefits for the coating industry, where the key factor is the ability to tailor both the energy and flux of the high fraction of ionized material present in a HiPIMS discharge. In this work, the spatial distribution of the HiPIMS discharge with positive pulses was investigated for different magnetron magnetic topology and negative/positive pulse configuration.

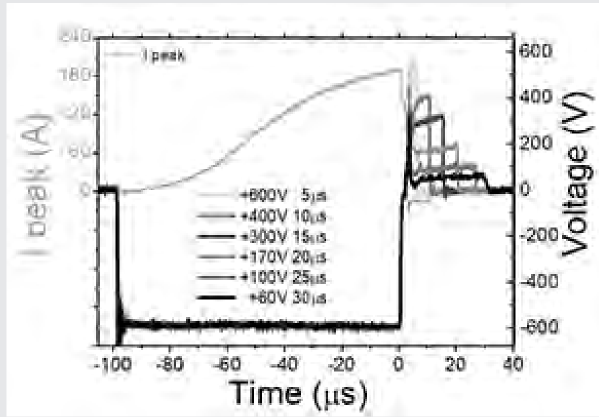

Figure. Discharge peak current and voltage waveforms for different voltage reversal configurations (ranging from +70 to +600V and from 5µs to 30µs)

Abstract 34

Design, preparation and investigation of tunable metal-dielectric coatings for plasmonic applications

Alexandr Belosludtsev, Deividas Buinovskis, Naglis Kyžas

Optical Coating Laboratory, Center for Physical Sciences and Technology, Savanorių ave. 231, Vilnius, 02300, Lithuania

***Corresponding author**
E-mail: Alexandr.Belosludtsev@ftmc.lt

Plasmonic resonance and devices based on this effect has received attention for the past few decades. They are finding applications in sensors, optical switches and semiconductor lasers, fluorescent spectroscopy, non-contact temperature-monitoring and other. Recently [1], we have developed the method the ultrathin continuous metal film deposition and precise monitoring. That gives a possibility of a precise control of the manufactured coatings with metal layers. This method was applied for fabrication of multilayer coating for beam splitters applications [2]. To continue this research, it was done the tunable plasmon-based device comprises a dielectric mirror and thin metallic layer. It was investigated the possibilities to tune the

spectral properties by adjusting the coatings structure. Dependencies on the mirror and metal layer changes were separately investigated. Such tuning gives the possibility precisely adjust the absorption peak position. Moreover it is possible to regulate not only the intensity, but the width of the peak. Multilayer coatings were prepared by the magnetron sputtering technology.

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Abstract 35

Gas Flow Sputtering of AlNx Thin Films as a High Temperature Strain Gauge

Jose Rivera

Fraunhofer IST, Bienroder Weg 54E, 38108 Braunschweig, Germany

***Corresponding author**
E-mail: jose.rivera@ist.fraunhofer.de

Traditionally, Aluminum nitride, a wide bandgap semiconductor (Eg=6.2 eV), has been used in applications for its strong piezoelectric response. The piezoresistive response of this film, however, has yet to be thoroughly investigated. Aluminum nitride thin films were reactively deposited onto bulk polished alumina as well as oxidized silicon wafer substrates with the goal of investigating its potential as a high temperature strain gauge. Gas Flow Sputtering was chosen as a deposition method due to its ability to maintain a very stable reactive process. The adjusted deposition parameters were the nitrogen gas flow rate and the applied pulsed negative substrate bias. Nitrogen flow was varied between 5 and 200 sccm, while the applied substrate bias was varied between xx and yy V. Under this range of conditions, both stoichiometric and non-stoichiometric aluminum nitride films were realized, but all investigated films showed well pronounced XRD patterns of the hexagonal AlN phase. Chemical compositions were investigated as well as film morphologies, electric and crystallographic properties. The changes in film properties were determined to be very strong for relatively small changes in nitrogen gas flow. Films deposited with a nitrogen gas flow between 10 and 200 sccm were found to be transparent, insulating and displayed consistent patterns of columnar growth structures. Nitrogen gas flows between 5 and 10 sccm, however, yielded films of a metallic appearance, resistivities ranging from 1x10-2 to 1x103 Ω cm and progressively displayed diminishing patterns of columnar growth with decreasing nitrogen film content. Film resistivity increased with decreasing substrate bias, and the piezoresistive response was shown to be more pronounced for higher resistivity films. The post deposition resistivity of this film, therefore, is indicative of the resulting piezoresistive behavior. The largest gauge factor (GF) achieved was 1.89 and was realized under 5 sccm of nitrogen gas flow and a xx V substrate bias. The measured temperature coefficient of resistance (TCR) of this film at 5 sccm was determined to be -2498 ppm/°C and saw little to no change between different applied substrate Biases. Gauges fabricated under such conditions, therefore yield piezoresistive properties characteristic to a metallic strain gauge, while at the same time exhibiting a characteristic semiconductor TCR. It was, however, shown that a piezoresistive response can, indeed, be realized with this film and that its strength can be manipulated by adjusting deposition parameters, making aluminum nitride a potential candidate for piezoresistive applications.

Abstract 36

Plasma Emission monitor for controlling the ion to neutral ratio and stoichiometry of HIPIMS processes

H. Gerdes[1], J. Rieke[2], R. Bandorf[1], T. Schütte[3], M. Vergöhl[1], G. Bräuer[1]

1 Fraunhofer IST, 2 IOT TU-Braunschweig, 3 PLASUS GmbH

***Corresponding author**
E-mail: holger.gerdes@ist.fraunhofer.de

High Power Impulse Magnetron Sputtering is a great technology for influencing the film growth by a higher amount of ionized species within the plasma in comparison to DC sputtering. When introducing a reactive gas into the deposition process not only the film composition is changed, but also the ion to neutral ration of the plasma. Therefore, this presentation will show a plasma emission monitor based control system, for keeping the ionization and the working point of the plasma constant. The ionization is kept constant by measuring the peak current and changing the charging voltage. The stoichiometry of the films is controlled by measuring the intensity of the emission line of the metal and controlling the oxygen within the sputtering chamber. These feature will be demonstrated with Ti in pure Ar and Ar/O2 atmosphere by deposition rates and the transparency of the films.

Abstract 37

Surface processes of energetic metal ions on HiPIMS target materials analysed by ion beam sputtering

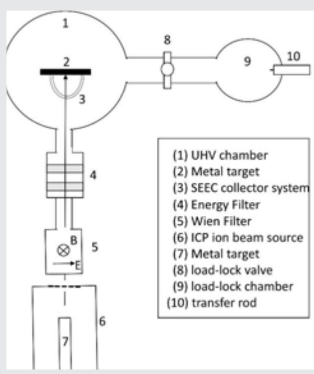
R. Buschhaus[1], M. Budde and A. von Keudell

Experimental Physics II, Ruhr-University Bochum, Germany

***Corresponding author**
E-mail: Rahel.Buschhaus@rub.de

In glow discharges the generation of secondary electrons at surfaces play an important role for the ignition and maintenance of the plasma. The ion-induced secondary electron emission coefficient (iSEEC) γ depends on the chemical state of the surfaces and is defined as number of released secondary electrons per incident ion. The coefficient is crucial for understanding HiPIMS discharge characteristics as this coefficient determines surface processes and thus effects the discharge regime (metallic and reactive mode) [1]. Of special interest is γ of multiple charged metal ions since these ions strongly influence self-sputtering effects [2]. Moreover, iSEEC is an important input parameter for many plasma simulations. The coefficients of some metals are already published in literature and measured by different techniques [3,4,5]. As the applied techniques basically measured directly in plasma, the determination of the iSEECs was rather indirect though. Moreover, energy dependence is mostly missing. However, the outstanding potential of the here presented experiment is the feasible analysis of surface processes induced by single and multiple ionized metal ions and their oxides within a broad energy and mass range.

The study of iSEEC is performed in an ultra-high vacuum (UHV) particle beam reactor (base pressure 10-5 Pa) (Fig.1). The experiment allows for separately analyzing the influence of different metal ions and their oxides on typical HiPIMS target materials. The applied metal ion beam source is based on an inductively coupled plasma (ICP) discharge which is fed with argon. A biased metal target inside the ICP is sputtered and generates metal atoms and ions. The particles are extracted from the ICP, pass a Wienfilter and are decelerated to a set energy. Thus, single and multiple ionized metal ions can be chosen. This ion beam with mass and energy selection is sent to a metal target surface in the vacuum chamber, where sputtering takes place and the iSEECs are computed. For both sputter targets, inside the ICP and the vacuum chamber, aluminum, copper and titanium is used. In addition, the chamber is equipped with a capillary atomic oxygen beam source, which enables the analyses of surface processes with oxides. For determination of the iSEECs a collector system, based on current measurements, is used as already described in [6].



We present first results and outline the potential of the ion beam experiment. We show iSEECs in dependence of the ion energy of commonly used HiPIMS target materials (Al, Ti, Cu).

Fig. 1. Schematic top view of particle beam experiment.

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List of authors

A

Aleksic, D.	Arthur Klink GmbH | DE
Amelal, T.	Leibnitz Institute Of Surface Engineering | DE
Arslan, H.	University of Catvia| LT
Arunprabhu, A.	Sheffield Hallam University | UK
Azens, A.	University of Catvia| LT

B

Bandorf ,R.	Fraunhofer Institute for Surface Engineering and Thin Films IST | DE
Banghard, M.	University of Tübingen | DE
Bárta, T.	University of West Bohemia | CZ
Batkovà, S.	University of West Bohemia | CZ
Beghi, M.	Politecnico di Milano | IT
Bellido, V.	Gencoa | UK
Belosludtsev, A.	Center For Physical Sciences and Technology | LT
Bernatova, K.	Masaryk University | CZ
Betiuk, M.	Institute of Precision Mechanics | PL
Bikse, L.	Institute of Solid State Physics | LT
Bowden, M.D.	University of Liverpool | UK
Bradley, J.W.	University of Liverpool | UK
Bräuer, G.	Fraunhofer Institute for Surface Engineering and Thin Films IST | DE
Breilmann, W.	Ruhr University Bochum| DE
Brenning, N.	Université Paris-Saclay | FR
Budde, M.	Ruhr University Bochum| DE
Buinowskis, D.	Center For Physical Sciences and Technology | LT
Bundesmann, C.	Leibnitz Institute of Surface Engineering | DE
Buschhaus, R.	Ruhr University Bochum| DE
Butler, A.	Université Paris-Saclay | FR
Butoi, B.	National Institute for Laser Plasma and Radiation Physics | RO

C

Cada, M.	Academy of Sciences of the Czech Republic | CZ
Capek, J.	University of West Bohemia | CZ
Cerstvy, R.	University of West Bohemia | CZ
Chen, Y.-H.	Feng Chia University | Taiwan
Chen, C.-M.	Feng Chia University | Taiwan
Chen, S.-Y.	Feng Chia University | Taiwan
Chen, T.-H.	Feng Chia University | Taiwan
Cikvaidze, G.	Feng Chia University | Taiwan
Corbella, C.	University of Latvia | LV
Ciperová, Z.	University of West Bohemia | CZ
Conti, C.	Politecnico di Milano | IT

D

Dellasega, D.	Politecnico di Milano | IT
Domanowski, P.	University of Science and Technology | PL
Dubau, M.	HVM Plasma Spol S.R.O. | CZ

E

Ehiasarian, A. P.	Sheffield Hallam University | UK
Eichenhofer, G.	4A-PLASMA | DE
Engler, T.	State Material Testing Institute Darmstadt | DE

F

Feder, R.	Leibnitz University of Surface Engineering | DE
Fekete, M.	Masaryk University | CZ
Fernandez-Martínez, I.	Nano4Energy SL; hip-V AB | SP; SWE

G

Gabriel, H.	PVT Plasma and Vacuum Technology GmbH | DE
Gabrusenoks, J.	Institute of Solid State Physics | LV
Gajewski, W.	TRUMPF Huettinger | PL
Gathen, V. S.-v.	Ruhr University Bochum | DE
Ganciu, M.	National Institute for Laser, Plasma and Radiation Physics | RO
Gerdes, H.	Fraunhofer Institute for Surface Engineering and Thin Films IST | DE
Groza, A.	National Institute for Laser, Plasma and Radiation Physics | RO
Gudmundsson, J.F.	KTH Royal Institute of Technology | SWE

H

Hajihoseini, H.	University of Iceland | IS
Haviar, S.	University of West Bohemia | CZ
Held, J.	Ruhr University Bochum| DE
Hnihica, J.	Masaryk University | CZ
Houska, J.	University of West Bohemia | CZ
Hovespian, P. E.h.	Sheffield Hallam University | UK
Hrebik, J.	Kurt J. Lesker Company | UK
Hsieh, P.-Y.	Feng Chia University | Taiwan
Hubicka, Z.	Academy of Sciences of the Czech Republic | CZ

J

Jaros, M.	PlasmaSolve Company | CZ
Jelínek, P.	University of West Bohemia | CZ

K

Kaestner, P.	IOT University of Technology | DE
Keudel, A. v.	Ruhr University Bochum | DE
Khan, I.	Sheffield Hallam University | UK

List of authors

Kipp, C.	IOT University of Technology | DE
Klein, P.	Masaryk University | CZ
Kolenaty, D.	University of West Bohemia| CZ
Kozak, T.	University of West Bohemia | CZ
Krbal, M.	University of Pardubice| CZ
Kyzas, N.	Center for Physical Sciences and Technology | LT

L

Lundin, D.	Université Paris–Sud, Université Paris–Saclay | FR

M

Marek, A.	HVM Plasma Spol.S.R.O. | CZ
Mares, P.	HVM Plasma Spol.S.R.O. | CZ
Martinez, I.F.	Nano4Energy SL and hip-V Joint Venture | SP
Mihalcea, B.	National institute for Laser, Plasma and Radiation Physics | RO
Minea, T. M.	Université Paris–Sud, Université Paris–Saclay | FR
Mirani, F.	Politecnico di Milano | IT
Moser, G.	J. Schneider Elektrotechnik | DE
Mroczynski, R.	Warsaw University of Technology| PL
Münz, W.-D.	Arthur Klink GmbH | DE
Musil, J.	University of West Bohemia | CZ

O

Obrusnik, A.	PlasmaSolve Company | CZ
Oechsner, M.	State Material Testing Institute Darmstadt | DE

P

Pajdarová, A.D.	University of West Bohemia | CZ
Passoni, M.	Politecnico di Milano | IT
Purans, J.	University of latvia | LV
Puzniak, M.	TRUMPF Huettinger | PL

R

Raadu, M. A.	School of Electrical Engineering, KTH–Royal Institute of Technology | SW
Rezek, J.	University of West Bohemia | CZ
Rieke, J.	Institute of Surface Technology, Braunschweig University of Technology | DE
Rivera, J.	Fraunhofer Institute for Surface Engineering and Thin Films IST | DE
Różański, P.	TRUMPF Huettinger | PL
Ryan, P.J.	University of Liverpool | UK

S

Santiago, J. A.	PVT Plasma and Vakuum Technology GmbH | DE
Scheerer, H.	State Material Testing Institute Darmstadt| DE
Schütte, T.	Plasus GmbH | DE
Schweiger, M.	J. Schneider GmbH | DE
Shukla, K.	Sheffield Hallam Universtiy | UK
Spemann, D.	Leibnitz-Institute of Surface Engineering | DE
Steins, H.	University of Tübingen | DE
Sugumaran, A.	Sheffield Hallam University | UK

V

Valizadeh, R.	STFC Daresbury | UK
Vasina, P.	Masaryk University | CZ
Vavassori, D.	Politecnico di Milano | IT
Vergöhl, M.	Fraunhofer Institute for Surface Engineering and Thin Films IST | Germany
Vlček, J.	University of West Bohemia | CZ
Vyskocil, J.	HVM Plasma Spol. S.R.O.| CZ

W

Walk, F.	University of Liverpool | UK
Wennberg, A.	Nano4Energy SL; hip-V AB | Spain; Sweden

Z

Zelechowski, M.	TRUMPF Huettinger | PL
Zikan, P.	PlasmaSolve Company | CZ
Zubkins, M.	Institute of Solid State Physics | LV

Conference committees

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Thin Films, Braunschweig, DE

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Local Organisation:

Network of Competence INPLAS

Network of Competence INPLAS e.V.
Bienroder Weg 54 E
38108 Braunschweig | GERMANY
www.inplas.de