HIPIMS 2015

6th International Conference on HIPIMS

BOOK OF ABSTRACTS

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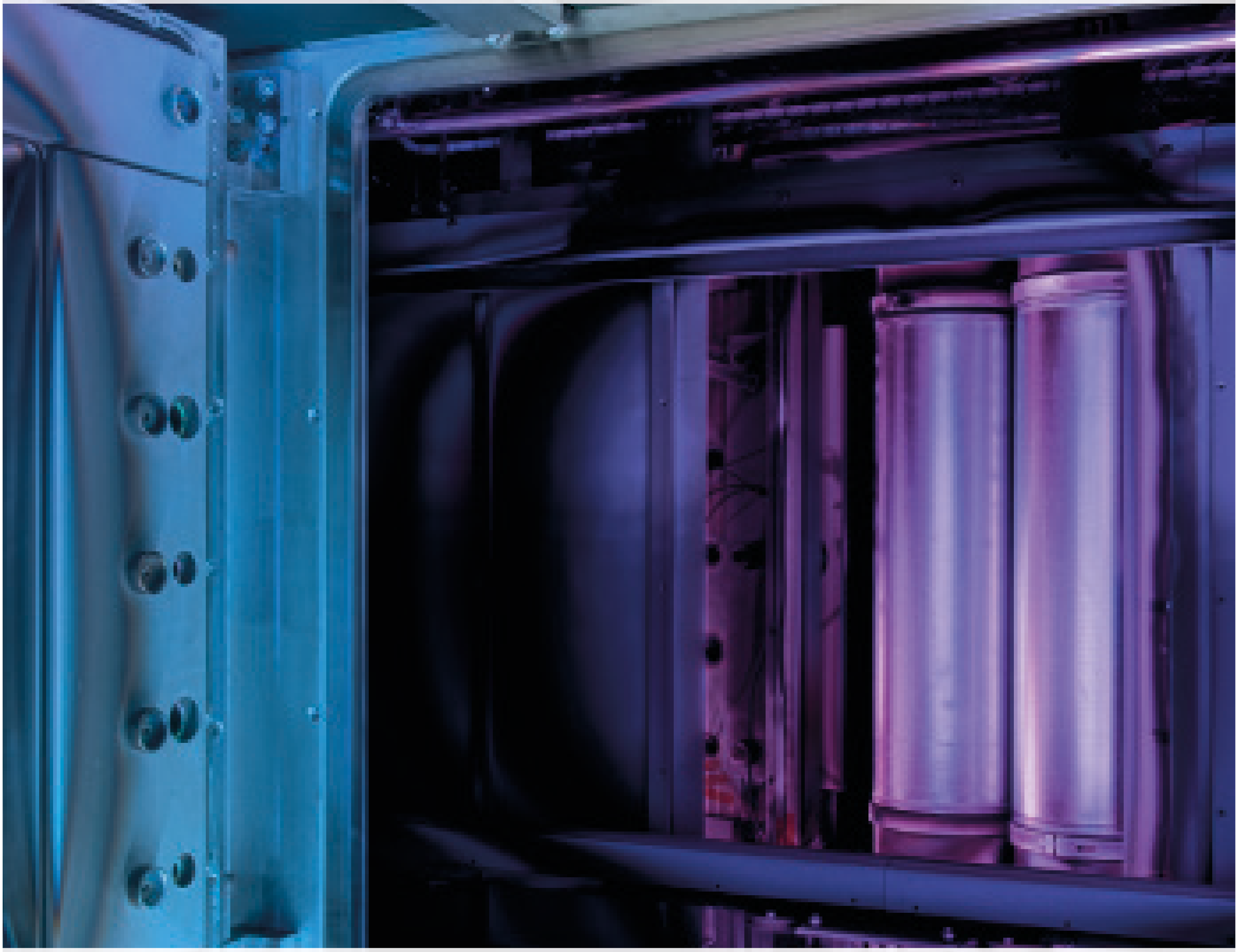

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Preface

Thin film technology and surface engineering are nowadays key components for numerous innovative products like efficient windows, flat screens, sensors or hard coatings used in tool coating and automotive applications, as well as products for everyday life. In line with the demands of surface technology, coating technology is also evolving and improving. The latest major technology jump was the introduction of pulse technology in physical vapor deposition.

High power impulse magnetron sputtering is the most recent development of pulse sputtering. After 15 years of intense academic investigation and development we observe today an increase of industrial processes and applications benefitting from this new technology.

As well as several international activities the international conference on fundamentals and applications of HIPIMS continues the success story of the HIPIMS days, initiated in 2004 at Sheffield Hallam University, UK. Becoming the only international conference especially dedicated to HIPIMS the HIPIMS conference is a venue for industrial and academic exchange on the latest developments in this fast evolving new technology. As a joint undertaking of Sheffield Hallam University SHU, Network of Competence for Industrial Plasma Surface Technology INPLAS, and Fraunhofer Institute for Surface Engineering and Thin Films IST the HIPIMS conference was launched in 2010 in Sheffield, UK.

With approximately 150 delegates the impact of the conference is underlined. The growing importance of HIPIMS technology was connected with a growth from initially around 100 participants to more than 150 participants during the last five years. Indication of the high impact towards applications is the composition of equal shares of participants from research and development (university and research institutes) and industry. Being a global conference representatives from over 25 different countries from all continents usually attend the meetings.

This year's conference focuses on the latest developments in academia and industry. Showing the impact of HIPIMS technology towards industrial applications presentations of industrial applications and products in the field of medical implants, semiconductor, hard coatings, as well as recent developments in power generation will be presented.

Significant increase in activities and high impact is found in the field of reactive HIPIMS. Solutions for reactive process control will be presented. From the material side different results on oxides, carbides and nitrides will be presented. The more academic contributions will focus on fundamental aspects of HIPIMS technology and recent results on modelling.

Dr. Ralf Bandorf and Prof. A. Ehiasarian
(Conference Chairman and Co-Chairman of HIPIMS 2015)

Contents page

HIPIMS 2015
6th International Conference on HIPIMS

Contents page

Contents page

Contents page

ORAL PRESENTATION

Development of nanostructured CrN/NbN coatings for medical prosthesis using HIPIMS

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CoCrMo alloy has a long history of successful use for the manufacture of medical devices due to its unique combination of mechanical, tribological and corrosion properties. Despite this, the alloy does contain ions which are sus-pected to trigger an allergic reaction in a small number of patients.
To address this application a nanoscale multilayer structured CrN / NbN coating was developed. The novel HIPIMS tech-nique has been used to pre-treat the substrate and deposit a dense coating. Thus the synergy between smart materials, (Nb is recognised for its biocompatibility and electrochemical stability and Cr for its tribological behaviour), unique coating structure (controlled on a nanoscale range) and advanced deposition method, (providing ionised plasma conditions for the coating growth) has been successfully exploited to produce a high quality application tailored coating.

LAXRD and TEM analyses revealed coating nanoscale multilayer structure with bi-layer thickness of 3.5 nm. The coatings were deposited with high thickness uniformity on real Co-Cr hip implants. Characterisation of CrN / NbN coated Co-Cr samples revealed high adhesion critical load values of Lc= 50 N in scratch adhesion tests and class HF1 in Daimler- Benz Rockwell C indentation tests thus out-performing a range of other PVD coatings deposited on the same substrate material. Nanoindentaton tests showed high hardness of 34 GPa and Young's modulus of 447 GPa. Low coefficient of friction (μ= 0.49) and coating wear coefficient, Kc (4.94×10^{-16} m^3 N^{-1} m^{-1}) were recorded in dry sliding tests.
In potentiodynamic polarisation experiments, the coatings showed excellent corrosion resistance outperforming other PVD Nb based coatings.
Metal ion release studies have been performed showing a reduction in Co release at physiological and elevated temperatures over a 28 day period to undetectable levels (<1 ppb) as compared to a peak of 22 ppb for uncoated samples.
Mechanical characterisation of coated components has been performed in order to assess the effect of the coating upon the substrate materials. Rotating beam fatigue testing in accordance with ASTM F1160 (2014) was performed, showing a significant increase (T test $P < 0.001$) in fatigue strength from 349 ± 59 MPa (uncoated) to 539 ± 59 MPa (coated).
Orthopaedic components were also tested to physiological load levels and exceeded the expected load requirements and those stipulated in ASTM F1800.
In vitro biological testing has been performed in order to assess the safety of the coating in biological environments, cytotoxicity, genotoxicity and sensitisation testing have been performed, all showing no adverse effects. The technology has been successfully transferred to industry for large scale production.

ORAL PRESENTATION

HIPIMS in Full Face Erosion Circular Cathode Semiconductor Applications.

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One of the great hopes for HIPIMS applications was their potential use and adoption by the semiconductor industry. Production of highly ionised particles has been of interest for deep in-trench filling as ionised particles can be directed by an electric field down the deep trench. However the technological reality is that the semiconductor industry has not been enthusiastic about this approach. The established

technologies exist for a good reason and any new technology has to be proven beyond a doubt before being adopted.

With regards to magnetron sputtering for semiconductor applications, the market is clearly dominated by circular sources where the magnetic field trap rotates in order to produce a clean target (Full Face Erosion, or FFE) as well as to maintain properties such as the uniformity and fill coverage from bump to valley.

There are several interesting phenomena taking place when a moving plasma and a high power pulsed plasma are combined. Both mechanisms have a very strong effect on the electric field map and offer both the challenge and the possibility of using strong transient electric field variations as part of the control mechanism of the discharge.

The current paper will focus on the characterisation of a 300 mm diameter FFE cathode unit adapted for use with a high pulsed power generator. Comparative data on DC and HIPIMS mode will be provided with regards to deposition distribution, coating properties, defects and trench filling.

ORAL PRESENTATION

Advanced coating architectures based on HiPIMS and Arc: High Ionization Triple

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Advanced coating designs are often done either by arc evaporation or by HIPAC (HiPIMS) sputtering process to adjust functional properties. However the combination of the two high ionized deposition processes to generate multilayer, nanomultilayer and nanocomposite coatings opened new horizons in tailoring of coating architectures. The arc evaporation itself is limited to specific cathode material properties (mostly metal alloys). HIPAC magnetron sputtering processes can be used to atomize and ionize materials which are difficult to evaporate or not evaporable by cathodic arc. The HIPAC magnetron specific materials are used both in the hybrid phase and for deposition of a functional top layer. Selected process features of the hybrid process, coating architectures and application results will be presented.

ORAL PRESENTATION

Designing the HiPIMS Process for Cutting Tool Coatings

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Metal cutting coatings should feature good adhesion and low intrinsic stress. Today's high film thickness values of 6 μ or even 10 μ for cutting inserts illustrate the demand for new methods of minimising the coating stress. This paper will discuss the interaction of the sputtering sources and the table Bias for the HiPIMS process with regard to this requirement. A new process design will be introduced that makes full use of HiPIMS at a Bias voltage as low as possible.

Using a dedicated HiPIMS Bias supply with synchronisation of the cathodes and the table is the key for achieving this. SEM images and data from nano-indentation such as microhardness and Young's modulus show the advantage of this new HiPIMS process design.

Machining tests reveal the correlation between the improved ratio of hardness to toughness and enhanced metal cutting properties in practical applications.

Keywords: HiPIMS, Sputtering, Cutting Tools.

ORAL PRESENTATION

High quality hard coatings produced by S3p™

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High performance hard coatings are produced to a great extent by PVD processes. In particular by a reaction of the metal flux with reactive gases like nitrogen, oxygen or hydrocarbon gases.

S3p™ (Scalable Pulsed Power Plasma) is the ideal technology to provide ionized material flux in order to produce dense, smooth and durable coatings.

Important features of S3p™ are power pulses with a density of 500 W/cm^2 to 2000 W/cm^2 and a rectangular pulse shape. The pulse duration can be adjusted independently of the pulse power level, within the range from 0.05 ms to 100 ms.
Reactive sputter processes with no hysteresis and therefore very high stability and reproducibility can be provided.

A new coating generation, BALIQ™, was recently introduced to the market, offering specific solutions in cutting applications with outstanding performance.

An overview of coatings and applications implemented by S3p™ will be presented.

ORAL PRESENTATION

HiPIMS power supplies – challenges and development

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Due to the high ionization efficiency of the sputtered target elements the High Power Impulse Magnetron Sputtering (HiPIMS) technology is winning interest and acceptance for industrial scale production of films with superior microstructure and properties. In order to deliver required very high instantaneous power density (up to 10 kW cm^{-2}) to the magnetron, a power supply exhibiting a long-time stability and reproducibility of working parameters is required. For a successful application on the industrial scale the power supply should also support the efforts made to surmount the main drawback of the HiPIMS processing, namely the lower deposition rate, as compared to DC magnetron sputtering.

In our contribution the working parameters of the new water-cooled TruPlasma Highpulse Series will be reviewed in order to give a deeper insight into the latest developments and functionality of the up-to-date commercially available HiPIMS power supplies and their influence on the HiPIMS plasma parameters. Furthermore, power supply features enabling tuning the process deposition rate as well as ensuring fast arc suppression will be discussed using experimental data.

HIPIMS 2015
6th International Conference on HIPIMS

ORAL PRESENTATION

Deposition of rutile TiO_2 films by pulsed and high power pulsed magnetron sputtering

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Thin films of titanium dioxide (TiO_2) are important high refractive index coatings in optical multilayer stacks. Especially interesting is the rutile crystalline phase of TiO_2 films because it's refractive index in the range of 2.6…2.7 is the highest available refractive index of materials being transparent in the visible and near infrared wavelength region. Rutile TiO_2 films in optical interference coatings therefore in principle allow achieving the desired optical function with a lower number of layers and a simpler design compared to alternative high index materials.

The crystalline growth of TiO_2 layers however usually starts with an amorphous underlayer of typically 40nm thickness before going over to an anatase or rutile crystalline structure. The amorphous underlayer typically has a refractive index in the range of 2.4…2.5. This significant difference to the index of the rutile film leads to a typical gradient in refractive index of TiO_2 single layers and makes application of TiO_2 films in precision optics practically difficult. The paper explores the possibilities of obtaining rutile TiO_2 films right from the beginning of film growth by significantly increasing energetic ion bombardment of the growing film and adatom mobility. The influence of a variety of process parameters such as substrate temperature, deposition pressure, magnetic field strength and rf bias is investigated.

The main focus of investigations was the comparison of a standard pulse process, a pulsed process with significantly increased pulse current and pulse process in genuine high power pulsed (HPPMS) mode.

Film deposition was done by stationary sputtering using the Double Ring Magnetron DRM 400. For pulse powering the pulse unit UBS of Fraunhofer FEP and the TruPlasma Highpulse 4006 of Hüttinger / Trumpf were used. Film characterization was done by XRD as well as by spectroscopic ellipsometry and photometry. Total optical losses were characterized by cavity ring-down spectroscopy (CRD) and absorption was measured by laser induced deflection. Results show that in a rather narrow range of process parameters the desired crystalline growth of TiO_2 with the rutile phase throughout the film can be obtained. This parameter set includes both the pulsed process with significantly increased pulse current and the genuine HPPMS mode, but in all cases requires a combination with substrate heating, rf substrate bias and adapted process pressure. A significant reduction of crystallite size and scattering losses was achieved by adding a small amount of silicon dioxide (SiO_2) to the film.

ORAL PRESENTATION

Pulse length and frequency control of target poisoning in reactive HiPIMS: application to amorphous HfO_2

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In conventional reactive magnetron sputtering, target poisoning frequently leads to a process instability that requires feedback control of the reactive gas flow rate to maintain a constant composition of the deposited layers. Herein, we demonstrate that effective control of the pulse length and / or frequency in high power impulse magnetron sputtering (HiPIMS) eliminates the need for any feedback control. Using this approach, we achieve stable reactive magnetron sputter deposition of hafnium oxide from a Hf target. Tuning of pulse length and frequency can be used to effect a smooth transition from a poisoned target surface condition (low duty cycles) to a quasi-metallic target surface condition (high duty cycles). Selection of appropriate duty cycle enables optimization of the deposition rate and the deposition of stoichiometric amorphous hafnium oxide (HfO_2). A model is presented for the reactive HiPIMS process in which the target operates in a partially poisoned mode with a distribution of oxide on its surface that depends on the pulse length.

ORAL PRESENTATION

Oxide semiconductor thin film deposition by HIPIMS reactive magnetron sputtering.

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Semiconductor WO_3 and Fe_2O_3 thin films were deposited on glass substrates with well conductive transparent electrode FTO and on silicon substrates with metallic Pt electrode. A pulsed magnetron system with pure metallic targets was used for this purpose working in reactive mode with $Ar+O_2$ working gas mixture. The pulsed frequency was changed from 100 Hz to 50 kHz with a different pulsed power absorbed in the discharge. The maximum discharge current density in the pulse was $jD \approx 4$ A cm^{-2} so the high fraction of sputtered particles was ionized. It was already shown [1] that this high degree of ionization lead to semiconductor films with higher quality regarding light induced photocurrents in a photoelectrochemical measurement. The further improvement of semiconductor quality was achieved by the application of a pulsed modulated RF or MF voltage on the substrate in order to create a DC induced self bias. This DC bias can control the energy of ions hitting the substrate. The pulsed modulation of this bias was synchronized with magnetron pulses in order to generate this DC bias only after the beginning of the pulse and a short time after the end of active discharge pulse. RF voltage and RF current probes were implemented in order to measure RF current and voltage waveforms supplying the substrate. These measured waveforms were used for determination of the ion flux on the substrate by a method developed by Sobolewski [2]. The advantage of this method is the possibility of ion flux measurement also when the conductive electrode is coated by an insulating layer. The ion flux was measured with time resolution during the HIPIMS magnetron pulse. Magnitudes of these ion fluxes together with magnitudes of these induced DC voltages were correlated with induced photocurrents for the deposited films at these conditions. Some optimal conditions were found where the photocurrents have maximum values. The deposited films were also analyzed by XRD, Raman spectroscopy, SEM microscopy.

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ORAL PRESENTATION

Reactive HiPIMS pulse optimization strategies in fabrication of all-dielectric optical coatings

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The HiPIMS approach has been documented to be suitable for the fabrication of high quality optical coatings exhibiting excellent optical and mechanical properties. However, reactive HiPIMS discharges are prone to excessive currents and related high probability of arcing, which is one of the main reasons that hampers its general acceptance and deployment on the industrial scale. In this contribution we describe various practical approaches that allow for arc-free deposition of metal oxide films. In the present work we investigate three strategies for arc elimination in reactive HiPIMS: (i) the magnetic field strength control through the use of paramagnetic spacers in between the magnetron head and the target, (ii) the use of specific pulse configuration comprising a combination of high pulse repetition frequency and short pulse duration, and finally, (iii) the superimposition of high-frequency voltage ripples to HiPIMS impulses.

Employing some or all of these pulse management strategies allows one to obtain arc-free reactive HiPIMS discharges, as demonstrated for Nb, Ta, and even Si targets (50 and 100 mm in diameter). We also show that the optimized HiPIMS can be used for the deposition of highquality optical coatings, including multilayer intereference filters, when a stabilized hysteresisfree transition between metallic and compound sputtering modes is established. The presented optimization strategies can thus be considered part of good practice for judicious application of reactive HiPIMS.

ORAL PRESENTATION

Exploring the structure zone transition in energetic deposition of Cu thin films by modulated pulsed power magnetron sputtering

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An Ar / Cu modulated pulsed power magnetron sputtering (MPPMS) discharge process is experimentally investigated and numerically modeled, in order to explore the influence of the pressure on the internal plasma parameters as well as on the microstructure of the deposited thin films. Cu thin films are deposited by MPPMS at adjusted pressures ranging from 0.1 to 0.7 Pa. The surface morphology, grain size and orientation, and microstructure of the deposited thin films are investigated by scanning electron microscopy (SEM), transmission electron microscopy (TEM) and x-ray diffraction (XRD). A global plasma model is developed for MPPMS discharges based on a volume-averaged global description of the ionization region, with considering the loss of electrons by cross-B diffusion. By fitting the model to duplicate the experimental discharge data, the temporal variations of internal plasma parameters are obtained and used to estimate the parameters directly related to the thin film growth processes, which are adopted in the extended structure zone diagram (SZD), to describe their influence on the microstructure of the deposited thin films. The microstructure and texture transition of the thin films is well-explained by the extended SZD, suggesting that the primary plasma processes are properly incorporated in the model, and the extended SZD is appropriate for the MPPMS as a new energetic deposition technique.

ORAL PRESENTATION

Ionized fraction and plasma parameters investigation in HiPIMS with Ti, Al and C target

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Plasma parameters, such as electron density, effective electron temperature, plasma and floating potentials together with ionization of sputtered particles have been investigated in non-reactive HiPIMS processes of Ar / Ti, Ar /A l, and Ar / C. A time-resolved Langmuir probe technique and a recently developed gridless ion meter have been used in the measurements. The aim of this study has been to measure and estimate the absolute values of the ionized fraction of three different target materials, and to prove that the Langmuir probe technique is a useful tool to estimate trends and changes in the degree of ionization for different process conditions. To cover a range of commonly used discharge conditions we have carried out measurements for different process gas pressures, discharge current densities, and pulse lengths. The pulse current density has been ranging between 0.5 A cm^{-2} and 2.0 A cm^{-2}, the pulse length has been set to 100 µs and 400 µs and the working gas pressure has been 0.5 and 2.0 Pa. It is found that by increasing the current density from 0.5 to 2.0 A cm^{-2} there is a general increase of ne independently of target material and position in time with maximum plasma densities of about 1 × 10^{18} - 5 × 10^{18} m^{-3} above the target race track. Also the ionized flux fraction, measured by the ion meter, is increased when increasing the current density and reaches a maximum value of 78 % in the Al discharge.

The work has been supported by the Czech Science Foundation through project 15-00863S and by the European Commission under the 7th Framework Programme through the ‘Factories of the Future’ program, FP7-2013-NMP-FoF grant number 608800.

ORAL PRESENTATION

Cathode voltage and discharge current oscillations and spoke behavior during HiPIMS discharge

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Recent investigations of the non-reactive HiPIMS discharge using high speed imaging, current probes and optical spectroscopy revealed that plasma is not homogenously

HIPIMS 2015
6th International Conference on HIPIMS

distributed over the target surface, but it is concentrated in regions of ionization zones called spokes and drifting above the erosion racetrack [1, 2, 3]. The spoke rotation velocity was evaluated around ~10 km / s [2]. Similar spoke rotation speed was obtained from oscillations on both floating potential of the probe and collimated optical signal [3]. Further observations differentiated ionization zone shapes into triangular or diffusive shape depending on the second ionization potential of the target material with respect to the first ionization potential of Ar [4]. Apart from previously mentioned inhomogeneity, several observations of oscillations on both discharge current and cathode voltage were reported during the high current phase of HiPIMS pulse [5]. Usually these oscillations were attributed as peculiar generator effect.Recently it was reported that such oscillations indicate the spoke formation [6].

In our experiments, the periodic oscillations on cathode voltage and discharge current were observed with frequencies between 250 – 400 kHz for strict experimental conditions: pressure ranged from 0.3 to 2 Pa and discharge current higher than 225 A. No oscillations were present for the pressure overreaching 2 Pa. For given pressure the frequency of oscillations depends on actual discharge current independently on the applied voltage. The decrease of the oscillation frequency with increase of the discharge current was proved in all studied pressure range. Increasing the pressure the oscillations frequency increased.

The spokes presence and their behavior were studied by high-speed imaging. The spoke appearance was strongly dependent on the pressure. At very low pressure, the triangular spoke shape was well recognized but with increasing pressure the shape became diffusive. Overreaching 2 Pa there was no spokes recognizable. Camera enables to record two successive images from the same pulse with time delay of 3 µs. Spoke rotation velocity and frequency was derived from the spoke image shift in the pressure range 0.18 - 0.5 Pa, where the spokes were well distinguished. The spoke characteristic frequency was arbitrary determined as multiplication of number of spokes with rotation frequency and ranges from 77 to 178 kHz. Spoke characteristic frequency is in the same order of magnitude as observed oscillations on cathode voltage and discharge current, nevertheless they are not identical. Spoke characteristic frequency is 3 – 4 times lower than the frequency of the oscillations.

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ORAL PRESENTATION

Correlation between Ion Transport and Plasma Oscillations in DC and HiPIMS discharges

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Recent findings show that plasma oscillations are commonly found in magnetrons, regardless of the power supply and power levels obtained. We present a comprehensive investigation of the oscillation properties in terms of amplitude, frequency and rotation direction using the 12 flat probes, installed azimuthally around the circular magnetron.
The investigated discharge conditions encompass both DC and HiPIMS discharges with current density ranging from 0.5m A / cm^2 to 5 A / cm^2. The results exhibit a wide spectrum of frequencies ranging from 250 Hz to 200 kHz. When the flat probes are negatively biased, the ion saturation current is collected and measured. The correlation between the observed oscillations and the ions transported away from the target allows establishing a qualitative understanding of the ion transport for wide range of discharge currents in DC and HiPIMS discharges. The results are compared with the Hall parameter, a measure commonly used to evaluate the cross-field transport, reducing from 16 in DC discharges to values of around 2 in HiPIMS discharge.

ORAL PRESENTATION

High-current impulse magnetron discharge with liquid target

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Over the past two decades the increasing attention has been paid to the magnetron sputtering technologies with hot or melted target [1 – 3]. Magnetrons with melted liquid target (MLT) allow for reaching such temperatures, when the vapour pressure of cathode material itself is sufficient for sustaining the discharge. In this case the working gas becomes unnecessary. Advantages of this technique include high deposition rate, which cannot be reached in conventional magnetron devices with cold cathode, as well as high energy efficiency, improved target material utilization and significantly smaller amount of defects than is usually induced by the working gas particles.

Earlier we have demonstrated the stable regimes of stationary (dc) magnetron discharge operated in copper and silicon vapors [3, 4]. Particularly, the plasma parameters were investigated for MLT with copper cathode. Measured plasma density and electron temperature were $n \sim 10^{17}$ m^{-3}, $T_e \sim 3$ eV, correspondingly. Based on the experience in high-current impulse magnetron discharge (HCIMD), first developed in 1990s [5] (which showed the way to high-power impulse magnetron sputtering — HiPIMS), we have recently implemented the stable regimes of high-power impulse discharge operated on a melted target. The high voltage pulses (~ 1 – 20 ms) were applied over 2-kW-dc MLT discharge. In present contribution we report the results of preliminary investigations of electric characteristics (voltage and current) and plasma parameters (n, Te, Upl) for the discharge with liquid copper target depending on power load, magnetic field configuration and the working gas pressure. Just like the dc MLT, the impulse regime (IMLT) is stably operated in metal vapour without the working gas.

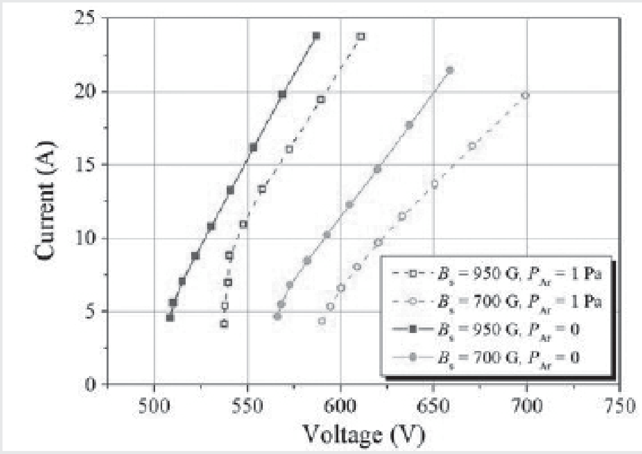

Figure 1: Current-voltage relations of a single IMLT pulse depending on surface magnetic field and Ar pressure

Figure 1 shows current-voltage relations during single IMLT pulse in working gas atmosphere as well as in vacuum for the same experimental conditions. One can see that closing the working gas inlet practically does not affect the discharge voltage and current. This observation indicates that the role of working gas in IMLT operation is insignificant. The measurements of plasma parameters and their spatial distributions (n, Te, Upl) in IMLT were conducted using the pulsed synchronized Langmuir probes. Particularly, plasma density measured 5 cm from the target surface is $n \sim 10^{19}$ m^{-3} that is 2 orders of magnitude higher than in case of dc MLT. Such values are typical for HCIMD (HiPIMS) regimes, however IMLT plasma naturally consists of target species only. IMLT is a promising technique for producing the high-quality coatings with high deposition rates.

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ORAL PRESENTATION

A Feedback Model of Magnetron Sputtering Plasmas in HIPIMS

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We present a 1-D feedback model that captures the essential elements of plasma pulse initiation and is useful for control and diagnostics of sputtering plasmas. Users can adjust many parameters to tailor the model to suit a specific scenario, including the secondary electron emission, ionisation probability, loss factors, and ion mass. It is expected the model will prove useful to the HIPIMS community, being robust and flexible enough to accurately model the physics of a variety of glow discharges, and providing insight into observed behaviours.

Our model falls into the class of single-species population models with recruitment and time delay, which show no oscillatory behaviour. The model can reproduce essential features of published time-current traces from plasma discharges and is useful to determine the key parameters affecting the evolution of the discharge. We include the external circuit and we focus on the time evolution of the current as a function of the applied voltage and the plasma parameters.

We find the necessity of a nonlinear loss term in the time-dependent plasma ion population to ensure a stable discharge, and we show that a higher secondary electron emission coefficient reduces the time delay for current initiation. We report that I-V characteristics in the plateau region, where it exists, fit a power curve of the form I = kV^n, where n is influenced most strongly by the nonlinear loss term.

ORAL PRESENTATION

A Model of Reactive HIPIMS Applied to Bipolar Dual Magnetron HIPIMS Deposition of Oxides and Nitrides of Ti

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A decrease in deposition rate is typically observed in metallic High Power Impulse Magnetron Sputtering (HIPIMS) compared to DC magnetron sputtering. Another decrease in deposition rate is typically observed after target poisoning during reactive sputtering. In reactive HIPIMS, a tendency to disappeared hysteresis have been reported. In this work we are focusing on all these phenomena, suggesting how they are interrelated. Experimental data on hysteresis during reactive HIPIMS of oxides and nitrides are reported and compared with a simplified model of reactive HIPIMS.

Oxide and nitride films of titanium have been deposited by reactive bipolar pulsed sputtering from pair of 2-inch round magnetrons. The bipolar HIPIMS regime was operated at pulse frequency in the range between 650 Hz and 10 kHz, at relatively short on-times and long offtimes. The mid-frequency operation featured high duty cycle with long on-times and short offtimes. Typical total average power was 300 W. To study the hysteresis phenomena, partial pressures were measured by mass spectrometry.

When the pulsing conditions have been gradually changed from mid-frequency operation to HIPIMS, the transition between metallic and reactive modes shifted towards lower oxygen flow due to lower sputtering rate, similar to decrease of deposition rate typically observed in metallic HIPIMS. Moreover, narrower or even disappearing jumps in pressure and voltage have been observed with HIPIMS for TiO_x. Compared to mid-frequency operation, the negative slope of O_2 sorption as function of O_2 partial pressure is clearly lower in case of HIPIMS resulting in lower critical pumping speed which implies disappearing hysteresis. Data on TiN_x are presented, too.

A model of reactive HIPIMS combining the Berg – type model of reactive sputtering with the global HIPIMS model of Christie has been presented recently [1]. The return probability of ionized sputtered metal has been selected as a parameter to quantify the degree of HIPIMS. The most important effect explaining the experimental data is covering of reacted parts of target by the returning ionized metal, effectively lowering the target coverage by reaction product at a given partial pressure. The model thus predicts less negative slope of sputtering yield and reactive gas sorption as functions of reactive gas partial pressure than in the DC or mid-frequency case.

The model exhibits good qualitative and quantitative agreement with experimental data for Ti oxides and nitrides. Moreover, the model predicts some interesting and technically relevant phenomena. When the sputtering yield of compound from the target is relatively high (factor > 0.2 to sputtering yield of metal, e.g. TiN to Ti), the deposition rate of reactive HIPIMS is always lower than in DC reactive sputtering for the same composition of deposited film. In contrast, when the compound sputtering yield is much lower (factor 0.0 < 0.10 to the sputtering yield of metal), higher deposition rate of reactive HIPIMS is predicted in the transition regime compared to DC reactive sputtering for some interval of deposited film composition. The deposition of Ti or Al oxides are examples of such materials.

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ORAL PRESENTATION

A parametric model for reactive high-power impulse magnetron sputtering

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Reactive magnetron sputtering has become a standard process for deposition of oxide and nitride films. Recently, the benefits of the deposition of oxide films using a controlled reactive HiPIMS process were demonstrated [1,2]. We have developed a parametric model of the reactive HiPIMS process, which helps us to understand the role of individual processes on the target and the substrate surfaces during the high-power pulses. The model calculates time-dependent compositions of the compound layers on the target and substrate surfaces in a pulse period, and the fluxes of metal atoms and reactive gas atoms and molecules onto the substrate, where the compound film is formed. The action of the discharge plasma is described by the target voltage and target current waveforms and by a set of internal discharge parameters which characterize the chemistry and the transport in the volume between the target and the substrate. The model takes into account specific features of the HiPIMS discharges, namely gas rarefaction in front of the sputtered target, backward flux of the ionized sputtered metal atoms and reactive gas atoms onto the target, and high degree of dissociation of reactive gas molecules in the flux onto the target and substrate. This phenomenological description of the discharge gives us a relatively simple model which allows us to quickly simulate reactive HiPIMS deposition under various process conditions.

In this work, we simulate the deposition of ZrO_2 films under conditions ranging from continuous dc sputtering to HiPIMS with the average target power density of 2 kW cm^{-2} in a pulse. We show that in the HiPIMS regime, the compound coverage of the target is significantly lower than in

the continuous dc sputtering regime at the same partial pressure of oxygen. This results in an increase of the deposition rate. However, enough oxygen must be supplied to obtain a stoichiometric compound on the substrate, which, we show, can be achieved by the increased ionization and dissociation of oxygen molecules in the HiPIMS discharge. The model calculations exhibit good qualitative agreement with experiments [2].

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ORAL PRESENTATION

Reactive high-power impulse magnetron sputtering of films – a process control and modelling

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High-power impulse magnetron sputtering (HiPIMS) with a pulsed reactive gas (oxygen) flow control was used for high-rate reactive depositions of densified, highly optically transparent, stoichiometric zirconium dioxide films. The depositions were performed using a strongly unbalanced magnetron with a planar zirconium target of 100 mm diameter in argon-oxygen gas mixtures at the argon pressure of 2 Pa. The repetition frequency was 500 Hz at the deposition-averaged target power density from 5 Wcm^{-2} to 53 W cm^{-2} with the duty cycles from 2.5 % to 10 %. Typical substrate temperatures were less than 130 °C during the depositions of films on a floating substrate at the distance of 100 mm from the target. Usual deposition rates, being around 10 nm / min, were achieved for the target power density of 5 Wcm^{-2}. An optimized location of the oxygen gas inlets in front of the target and their orientation toward the substrate surface made it possible to improve quality of the films due to minimized arcing on the sputtered target and to enhance their deposition rates up to 120 nm / min for a deposition-averaged target power density of 52 Wcm^{-2} and a voltage pulse duration of 200 µs [1,2].

To understand complicated processes during reactive HiPIMS of dielectric films, we have developed a parametric model. The model takes into account specific features of the HiPIMS discharges, namely gas rarefaction in front of the sputtered target, backward flux of the ionized sputtered metal atoms and reactive gas atoms onto the target, and high degree of dissociation of reactive gas molecules in the flux onto the target and substrate. Moreover, a local overfilling of the reactive gas in front of the reactive gas inlet is considered. The model makes it possible to calculate the time-dependent compositions of the compound layers on the target and substrate surfaces in a pulse period, and the number of metal atoms, forming the compound layers, which are deposited onto the substrate per second.

We used this model to clarify the experimental results achieved by us for the controlled reactive HiPIMS of zirconium dioxide films.

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ORAL PRESENTATION

Deposition carbon-based hemocompatible and biofunctionalisable coatings for cardiovascular applications using HiPIMS

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With economic development and the associated decline in communicable diseases and extended life expectancies, cardiovascular disease is now a leading cause of death worldwide. Coronary artery disease is the largest contributor to this epidemic. Implantable cardiovascular devices such as stents, pacemakers and heart valves are playing an increasingly important role in modern cardiovascular medicine. However, clinically effective and biocompatible synthetic materials for cardiovascular devices are currently lacking. This creates a scientific and clinical imperative to improve the efficacy of biomaterials available for vascular implantation and repair.

To be truly hemocompatible, materials need to address multiple issues simultaneously. Firstly they must have low thrombogenicity so as not to induce potential fatal blood clots, secondly, they should limit inflammatory responses which cause the infiltration and proliferation of smooth muscle cells that block blood vessels and ideally, they should encourage the overgrowth endothelial cells which form the natural lining of blood vessels. Contemporary cardiovascular stents are made from metals, such as stainless steel, to provide the mechanical strength needed to reopen and hold open blocked blood vessels. These materials are inherently thrombogenic so that patients need to be on blood thinning medication for prolonged periods, increasing their risk of life threatening bleeding episodes. Metal stents typically occlude due to the inflammatory response (in a process known as restenosis). This has led to the introduction of drug eluting stents, which unfortunately have the side affect of further delaying endothelialisation, leading to potentially fatal late stent thrombosis events. A new generation stent platform that maintains low thrombogenicity whilst limiting restenosis is needed.

In this work, we use HiPIMS to deposit carbon-based coatings. These coatings show dramatic reductions in thrombogenicity compared with stainless steel, comparable to the plasma activated coatings (PAC) [1,2], previously deposited using a plasma enhanced chemical vapour deposition process with energetic ion bombardment. We show also that, like PAC, these coatings enable the direct covalent immobilisation [3] of biological molecules on their surface. This opens up the potential of using inherently hemocompatible biomolecules [4], such as tropoelastin or perlecan, on the surface of a stent to inhibit restenosis and accelerate endothelialisation.

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ORAL PRESENTATION

Characterization of the mixed-mode carbon HIPIMS process

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Mixed-mode carbon HIPIMS uses a conventional circular magnetron to generate a highly ionized carbon plasma. In this process, the discharge current increases to the point where an arc ignites briefly on the surface of the target. The first studies of carbon mixed-mode operation identified nanometre-scale particles in the deposited films, and we are investigating the mixed-mode process with the aim of optimizing the formation of these particles as a route to nanodiamond synthesis. A second application of the process is the generation of ionized carbon beams for the deposition of sp^3-bonded carbon films.

We have investigated the mixed-mode regime by analysis of the discharge current and voltage, still photography, time-resolved spectroscopy, and AFM and SEM of deposited films. Mixed-mode operation significantly increases the ionized fraction of the carbon flux at the substrate compared to HIPIMS deposition without arcs. The short-lived arcs present in mixed-mode operation show similarities to a cathodic arc discharge. The arc spots move rapidly due to the retrograde $E \times B$ steering effect of the magnetron’s magnetic field, limiting the generation of macroparticles.

ORAL PRESENTATION

W-DLC coatings for industrial applications deposited by a combination of HIPIMS and unbalanced magnetron sputtering at low temperature

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Thin films of tungsten containing diamond-like carbon (W-DLC) possess superior mechanical, electrical and chemical properties and thus attract an interest from both scientific and industrial fields. Coatings composed of pure or alloyed W-DLC are typically characterized by high wear resistance and low coefficient of friction at the same time together with extraordinary hardness at high temperatures and resistance against corrosion. What is equally important, the adhesion of W-DLC films is typically very good due to low intrinsic stress level. However, a successful industrial application sets even higher demands on the adhesion of such a coating. The adhesion must be excellent even if the coating is deposited at a low temperature (200 °C at maximum) which is required by most types of steel substrates used in machine industry. The combination of HIPIMS and unbalanced magnetron sputtering (UBMS) can help to overcome this problem. HIPIMS itself is known to promote the adhesion of coatings and can be applied even for substrates requiring low temperatures. On the other hand, UBMS is suitable for deposition of well-defined structures. The combination of both techniques can be expected to result in coatings of finer structures and with extraordinary adhesion.

In this contribution, we present a study of the structure and properties of W–DLC coatings for industrial applications deposited by a combination of HIPIMS/UBMS at low temperatures (up to 200 °C). The films were sputtered from a WC target in a mixture of argon and acetylene (C_2H_2) on substrates from industrial bearing steel hardened to 60 HRC of Martensite and Bainite structures. The structure and properties of the coatings (roughness, adhesion, coefficient of friction, hardness and elastic modulus) were studied in the dependence on the amount of C_2H_2 in the working gas mixture and the bias voltage applied to the substrate.

ORAL PRESENTATION

Synthesis of Tetrahedral Amorphous Carbon by Mixed Mode HiPIMS Deposition

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Tetrahedral amorphous carbon (ta-C) is a technologically important form of carbon containing a high fraction of sp^3 bonded atoms. The key to synthesizing ta-C is the presence of energetic carbon ions with an optimal energy in the vicinity of 100 eV. Despite substantial efforts, conventional sputtering approaches based on DC operation or HiPIMS have struggled to produce ta-C due to insufficient ionization, and so ta-C is typically produced commercially using cathodic arc deposition. Here, we show that operating HiPIMS in mixed mode, in which controlled arcs ignite on the surface of the cathode, produces a sufficiently high ionization fraction to enable deposition of ta-C.

Films were deposited onto silicon substrates using a substrate bias between 0 and –100 V, pulse lengths between 90 and 210 µs and Ar pressures between 1.75 and 4 mTorr. Optimal conditions for ta-C production involved long pulse lengths, pressures no greater than 2.25 mTorr and the highest bias. At lower biases and higher pressures the sp^3 fraction was substantially smaller, reducing to as little as 13%. The sp^3 fraction was determined using XPS and confirmed independently using the plasmon peak in EELS measurements. Ellipsometry showed that Tauc optical gap is proportional to the sp^3 fraction, being as high as 2.7 eV for the ta-C films and 1.7 eV for the films with low sp^3 content. Other key signatures of ta-C were all present, including high compressive stress, a symmetric Raman peak and very low surface roughness as measured by AFM.

ORAL PRESENTATION

TiCN(H) nano-composites deposited by two gases reactive HiPIMS

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The aim of this study is to investigate the potentiality of an HiIPIMS reactive process with two gases, evaluating the process control and the properties of the deposited coatings compared to DC magnetron sputtering. Starting from TiC(H) HiPIMS coatings results, TiCN(H) nano-composites were deposited characterizing their composition, structure and mechanical properties in industrial conditions with no sample biasing at room temperature.

Titanium Carbonitride films were deposited in an industrial close field apparatus (target size 12” x 4.9”) by HiPIMS from a titanium target with C_2H_2 and N_2 reactive gases. Reactive gases flux was changed in order to obtain (C+N)/Ti ratios

in the coating from 1 up to 3. The reactive process was studied by OES and correlation of Ti and C content in the HiPIMS process can be related respectively to Ti 499 nm emission line and H(656nm) / Ti(499 nm) emission lines ratio. The C / N ratio is determined primarily by the nitrogen flow. The composition of the coatings was measured by EDX and the results confirmed an easy control within the ternary diagram by OEM.

XRD characterization showed the presence of TiN and TiCN nanocrystals, surrounded by an amorphous matrix, with size (3 – 30 nm) and spacing dependent on C content. The preferential (111) grains orientation and compressive macrostress were investigated for the different composition with pole figures. As C content increases in Raman spectroscopy the D and G peaks emerged, showing the formation of a C - C or C- N amorphous matrix.

The deposited coatings depending on composition showed by nanoindentation hardness in the range 10^{-35} GPa and elastic modulus from 150 to 300 GPa. A maximum was observed for stoichiometric TiCN films with N / C content around 0.6 - 0.4. The hardness values achieved are comparable to coatings in literature deposited by DC sputtering with biasing. Microscratch and tribotest showed a decrease of the friction coefficient as carbon content increases.

Keywords: HiPIMS, TiCN, nanocomposite, reactive sputtering

ORAL PRESENTATION

HiPIMS AlTiN COATINGS

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AlTiN is a promising anti-oxidation and corrosion protective material. Thanks to the formation of a coherent, compact and adherent alumina scale, it can be applied in harsh environments.

In this work, the intrinsic properties of AlTiN films produced by reactive High Power Impulse Magnetron Sputtering (HiPIMS) have been investigated. Different substrates were chosen considering their technological interest: a common stainless steel (AISI 304), the T91 martensitic steel, which is conventionally used for nuclear fusion and fission plants, and, finally, a light alloy for aeronautic applications (γ-TiAl), which represents a possibility to replace Ni based superalloy in aircraft turbine engines.

Working gas composition has been systematically changed (N_2 / (Ar + N_2) atomic ratio 10, 25, 50 ,75, 100 %). Morphology, microstructure, hardness, elastic modulus, residual stress have been studied through high resolution methodologies. Microstructural analyses were carried out by Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD). Moreover, films nano-mechanical properties, as hardness (H) and Young’s modulus (E), were estimated by nano-indentation tester, while adhesion was evaluated by scratch tests. In order to investigate the average residual stress in the produced coatings, a recently proposed approach was used, which involves an incremental FIB milling of annular trenches at material surface, combined with high resolution SEM imaging [1].

Finally, AlTiN/T91 coatings were subjected to long duration tests for the evaluation of corrosion by molten metal (Pd or Pb-Bi alloy), while all the other films were tested in air at high temperature, to investigate the behaviors of AlTiN films subjected to thermal cycles.

SEM and XRD results showed a significant modification of films' microstructure due to selected deposition parameters: it ranges from quasi-amorphous structure at high N_2 percentage to crystalline one at low nitrogen content. This means that the energy flux carried to the substrate considerably changes. Mechanical properties were found to be extremely promising: measured hardness reached 40 GPa. Moreover, by optimizing N_2 percentage during the deposition process, it is possible to maximize the H / E ratio, which provides an indicator of the coating's elastic strain at failure. Finally, it was found how microstructure and / or mechanical properties of the coatings help to foresee the film / substrate system adhesion.

The possibility to choose different experimental conditions coupled with an extensive characterization of the deposited coatings, allows to obtain useful information about structure / properties correlations and then, to optimize deposition parameters to achieve the required characteristics for specific applications.

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ORAL PRESENTATION

Influence of High Power Pulse Magnetron Sputtering pulse parameters on the reactive gas N_2 in the deposition process of (Cr, Al)N coatings

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In plastics industry the injection molding tools are subjected to adhesive and abrasive wear as well as corrosion. In this regard, (Cr,Al)N coatings deposited by physical vapor deposition (PVD) have a high potential to be used as protective coatings. The high power pulse magnetron sputtering (HPPMS) technology (also referred to as high power impulse magnetron sputtering, HiPIMS) offers several advantages with regard to the deposition of these coatings. Variation of the pulse length of 40 µs, 80 µs and 200 µs at constant cathode mean power of 5 kW, has a significant influence on the current-voltage-characteristic (I-U) of the cathodes, the chemical composition and the mechanical properties hardness and elastic modulus of the (Cr,Al)N coatings as well as on the reaction layer on the top of the (Cr,Al)N coating, which impacts the interactions between the coated tool and the plastics in terms of adhesion. The present work deals with the investigation of the influence of HPPMS pulse length on the reactive gas N_2 in the deposition process of (Cr,Al)N coatings. For this reason, the HPPMS plasma was analyzed at the substrate side via a retarding field energy analyzer (RFEA) and at the cathode by means of energy resolved mass spectrometry (MS). The RFEA was used to analyze the ion current densities at different HPPMS pulse length 40 µs, 80 µs and 200 µs as well as to investigate the ion energy distribution

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(IEDF) in dependence of the bias voltage. The MS was used to analyze the intensities of CrII, AlII, NII and N_2II in the HPPMS plasma. The results revealed that a decrease of the pulse length leads to an increasing ion current density and an increasing dissociation of molecular nitrogen, which has a significant influence on the chemical composition of the (Cr,Al)N coating and the reaction layer on the top. Based on these results it can be noted that it is possible to adjust the (Cr,Al)N coatings for plastics processing by variation of the HPPMS pulse parameters.

Keywords: PVD, CrAlN, HPPMS, HiPIMS, RFEA, mass spectrometry

ORAL PRESENTATION

Highly Ionized Deposition of CrN

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CrN is a well-known material in different industrial application providing good electrical conductivity, high hardness, corrosion resistance and high temperature stability. Recent developments in highly ionized deposition, i. e. high power impulse magnetron sputtering lead to significant improved properties and advanced productivity for industrial use.

This presentation will discuss the influence of different HIPIMS techniques on the adhesion, hardness, composition and crystal structure of the thin films. Therefore the gas composition (ratio between Ar and N_2), the working pressure were changed. While conventional processes require temperatures in the range of 250 °C and additional biasing the presented films showed hardness values up to 2900 HV without substrate bias or additional heating.

ORAL PRESENTATION

Target Poisoning in Mixed Ar, N_2 and CH_4 Atmosphere, in Processes Using Different Target Materials for HIPIMS / DC and DC Cathode Modes.

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Reactive sputtering in mixed Ar + N_2 + CH_4 atmosphere is a widely used industrial process however the poisoning of different target materials under different sputtering discharges in this complex environment is insufficiently described.

In mixed Ar + CH_4 + N_2 atmosphere, at low flow processes were influenced by methane whereas at high flow they were dominated by nitrogen indicating the formation of carbide and then carbonitride compounds. This was observed for both TiAl and V targets in DC as well as in HIPIMS mode.

Vanadium targets operating in DC mode were poisoned at 55 % of reactive gas flow. Poisoning resulted in a 2-fold increase in total pressure, a 50 % increase in discharge voltage/current ratio, a 5 fold drop in V(I) optical emission intensity and a 10 fold drop in V^+ and Ar^+ fluxes obtained from energy-resolved mass spectroscopy.

TiAl targets in DC mode poisoned at lower reactive gas flows and exhibited narrower hysteresis than V due to the higher reactivity of the target material. The voltage/current ratio of TiAl targets went through a minimum with a flow, while for V target it increased with flow.

For HIPIMS both targets poisoned earlier and the hysteresis was narrower than in DC mode. As confirmed by trends in the partial pressure, the voltage/current ratio and ion fluxes of metals and reactive gasses. These effects are due to higher reactivity of the plasma as evidanced by higher fluxes of N^+ and N_2^+ and radicals containing H, C and N. The voltage/current ratio reduced by 50 % as the target is poisoned in contrast to operation in DC mode where it increased. This could be attribiuted to efficient ionization and drop in plasma impedance.

Pathways for poisoning and resulting ion fluxes are discussed.

POSTER PRESENTATION

HiPIMS Deposition of Tungsten Trioxide Thin Films.

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Tungsten oxide is well known for its electrochromic and photocatalytic properties. Thins films of WO_3 were deposited using a single magnetron which was situated directly under the sampleholder. Process gas (Ar) at a flow rate of 20 sccm and reactive gas (O_2) at a flow rate of 8 sccm were let in the chamber trough 2 separated gas mixers in a shape of metal tube ring with perforations. Gas mixers are set in a space between the magnetron and the sampleholder. As a power supply for the magnetron an ADL Gmbh. DC unit with a MELEC Gmbh. SIPP2000USB plasma DC pulse power controller was used. During the cycles the pulse shape was monitored using an oscilloscope. There were series of samples created using different pulse shape forms and duty cycles from 0.3 % 'till 5 %, which indicate different sputtering modes, though it is worth to mention that conventional DCMS mode was also used for statistics sake.

Films were sputtered on a several substrates: glass, silicon wafer and NaCl crystal.
Acquired samples were studied using by XRD method in order to gain data on a thin film structural properties. NIR and Raman spectroscopy were used as a spectroscopic analysis methods.
During deposition we observed significant drop, up to 3 times compared to DCMS, in deposition rate, which effect was reported by Hemberg et al. [1] All samples in this study are amorphous, what was concluded from the XRD and Raman spectra. Thin films are transparent with common for tungsten trioxide spectra curve. Presence of terminal oxygen was confirmed by NIR.

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POSTER PRESENTATION

Investigation of discharge structure inhomogeneities observed in high-current quasi-stationary magnetron discharge

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Over the past 5 years several research groups have been observing and investigating a peculiar phenomenon in high-power impulse magnetron sputtering (HiPIMS) discharges, namely, so-called spokes or ionization zones (e. g. recent publications [1 – 3]). Considerable effort is being made to determine the mechanisms of inhomogeneities appearing in such plasmas, and to find a way to control their formation. Our present contribution deals with the investigations of similar effects taking place in quasi-stationary phase of high-current impulse magnetron discharge

(HCIMD) [4]. HCIMD is a high-voltage (up to 1.5 kV), high-current (up to 20 A / cm^2), long (~ 1 – 40 ms) impulse sputtering tool [4].
The HCIMD plasma was investigated using the synchronized fast gated camera (BIFO K011) and fiber optic spectrometer (Avantes AvaSpec-2048 × 14), both oriented end-on with respect to the cathode. Fast camera allows for taking nine images with programmable frame times (100 ns – 100 µs) and frame delays (100 ns – 100 µs) after triggering. The spatial resolution of the matrix is 340 × 340 pixel for each frame. The spectral sensitivity range is 400 – 800 nm. Camera was equipped with Zenitar-M 50 mm lens.

HCIMD was initiated by applying a high voltage pulse across the pre-ionized discharge region. Cu, Mo and Ti 90-mm-diameter disc targets were used.

The voltage-current characteristics were obtained for the range of transverse magnetic field on the cathode surface B_s = 0.06 – 0.15 T and working gas (Ar) pressures P_{Ar} = 0.5 – 2 Pa. Fast camera images were recorded throughout the pulses for all experimental regimes, and the regions of plasma inhomogeneities formation was found. Our observations of the initial stage of HCIMD indicate that in certain discharge current range there is a characteristic time (~ tens µs) needed for the structure of spokes to develop. The fast camera images were analyzed together with the optical emission spectra. We provide the discussion of observed plasma behavior and compare it to the case of HiPIMS studies.

We thank Prof. Yuri A. Lebedev (Topchiev Institute of Petrochemical Synthesis, RAS) for providing the fast camera for these experiments.

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POSTER PRESENTATION

Industrial Scale Deposition of Diamond-like Carbon Thin Films using Ne-based HiPIMS Discharge

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High power impulse magnetron sputtering (HiPIMS) has been successful in providing highly ionized deposition fluxes for most common metals (Cu, Al, Ti). However, it is challenged when non-metals such as carbon is considered. Highly ionized carbon fluxes (up to 100 %) are essential for the synthesis of diamond-like carbon and tetrahedral amorphous carbon thin films. Earlier reports have shown that the C^+ / C^0 ratio in HiPIMS does not exceed 5 % [1] and film densities and sp^3/sp^2 bond fractions are substantially lower than those achieved using ionized physical vapour deposition based methods such as filtered cathodic vacuum arc and pulsed laser deposition. In our previous work [2], we demonstrated that Ne-based HiPIMS discharge entails energetic electrons as compared to Ar-based HiPIMS discharge facilitating the generation of highly ionized C fluxes as well as diamond-like carbon thin films with mass densities in the order of 2.8 g / cm^3.

In this work, we perform industrial scale deposition of diamond-like carbon thin films using Ne- as well as Ar-based HiPIMS discharge. In order to investigate the effect of electron temperature enhancement and its correlation to generation of C^{1+} ion fluxes in Ne-based HiPIMS discharge, we perform time-averaged and time-resolved measurements of electron temperature as well as ion density at the substrate position using a flat probe. We also investigate the effect of plasma properties on the ionization of sputtered C as well as buffer gas species by measuring the optical emission from the discharge. In order to correlate the plasma and film properties, we synthesize C thin films under energetic deposition conditions and investigate structural (mass density, sp^3/sp^2 bond fraction, H content) and mechanical (hardness, elastic modulus, adhesion strength) properties of the resulting diamond-like carbon thin films.

Keywords: DLC, ta-C, carbon ionization, HiPIMS.

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POSTER PRESENTATION

Highly Ionized Gas Flow Sputtering of Alumina Coatings

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High Power Impulse Magnetron Sputtering HIPIMS has shown that the increased ionization has a beneficial effect on the growing films. Significantly improved coatings or completely unique properties can be achieved by HIPIMS. Gas Flow Sputtering GFS as a specifically hollow cathode process allows for high local deposition rates. Even though a high plasma density exists in the source the film forming species are thermalized and mainly neutral.
Applying pulsed power, as used for HIPIMS to a GFS source (Highly Ionized Pulse Plasma Gas Flow Sputtering – HIPP-GFS) leads to enhanced ion content of the film forming species. A reactive process of aluminum targets running with additional oxygen for alumina deposition will be investigated. The presentation will focus on the plasma properties of the HIPP-GFS determined by optical emission spectroscopy and flat probe measurements. The results will be correlated to film properties of alumina coatings.

POSTER PRESENTATION

Tailoring the microstructure and properties of CrN thin films by HiPIMS in Deep Oscillations Magnetron Sputtering (DOMS) mode.

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In the last decade, new magnetron sputtering deposition techniques have been developed to produce highly ionized fluxes of sputtered material and, hence, to allow an increased control over the energetic ion bombardment (energy and direction of the deposited species). Two of these recent developments are: High-power Impulse Magnetron Sputtering (HiPIMS), also known as High-power Pulsed Magnetron Sputtering (HPPMS), and Modulated Pulsed Power Magnetron Sputtering (MPPMS). Both HIPIMS and MPPMS deposition techniques are characterized by the application of very high target power densities to achieve higher plasma densities and subsequent ionization of the sputtered material. It has also been demonstrated recently that the highly ionized plasma generated by HiPIMS technique can be usefully utilized to tailor the microstructure and properties of magnetron sputtered CrN coatings.

Recently, a new design of the MPPMS process named deep oscillation magnetron sputtering (DOMS) was developed. The main objective of the present work was to tailor the microstructure and properties of CrN thin films. CrN thin films where deposited by DOMS. The energetic ion bombardment of the growing films was controlled by changing the peak power at two different deposition pressures (0.3 and 0.7 Pa). The structure, morphology and mechanical properties of CrN coatings were studied. For comparison, two thin films were deposited by DC magnetron sputtering

(DCMS). One at deposition pressure of 0.3 Pa with -80 V substrate bias, the other one at deposition pressure of 0.7 Pa without substrate bias. The same average target power was used in all depositions (1200 W), in order to minimize the thermal effects on the films.

The CrN deposited at low pressure (0.3 Pa) by DCMS and DOMS have dense morphologies and the same [200] preferential orientation. However, the hardness of the CrN films deposited by DOMS is higher (21 to 27 GPa as compared to 17 GPa for DCMS) while featureless morphologies are obtained at high Pp values instead of columnar microstructures. The DCMS films deposited at high pressure (0.7 Pa) have an open columnar porous morphology typical of thin films deposited under low energy bombardment and/or high atomic shadowing effect. The film has a low hardness (7.2 GPa), low Young modulus (159 GPa) and a [111] preferential orientation. The CrN films deposited at high pressure by DOMS have similar microstructures and properties as the ones deposited at low pressure. Ionization of the sputtered Cr species increases both their energy and impinging angle on the growing film surface, allowing to overcome the shadowing effect at high pressure. The deposition rate of films deposited by DOMS is substantially lower than the deposition rate of the films deposited by DCMS. For low values Ppa, the deposition rate for DOMS corresponds to between 40 and 45 % of the deposition rate of the film deposited by low pressure DCMS.

POSTER PRESENTATION

HIPIMS ITO films from a rotatable target for applications in strain gauges.

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High Power Impulse Magnetron Sputtering (HIPIMS) allows the deposition of thin films from plasmas with high metallic ion content. In this way, the energy provided by depositing metal ions influences the film growth and can lead to better or new film properties. At the Fraunhofer IST, HIPIMS has been successfully used to deposit indium tin oxide (ITO) films from planar ceramic targets. The use of rotatable cylindrical cathodes is also a promising option due to their many advantages: better material utilization, longer durability, reduced arcing and more. In this work, ITO films were produced from a cylindrical ceramic target aiming at future applications as thin film strain gauges. The films were heated during the deposition process and the influence of substrate temperature was investigated. The specific resistivity of the films was determined and their optical properties were analyzed in terms of the dimensionless extinction coefficient k. The resistivity as a function of strain was measured and temperature coefficients of resistance (TCR) were determined. After deposition, films were also annealed in vacuum and their electrical and optical properties were further investigated.

Keywords: HIPIMS, indium tin oxide, ITO, rotatable, cylindrical, cathode, target, strain gauge, electrical, optical, properties.

POSTER PRESENTATION

Superconducting Cavities Nb coating with biased HiPIMS technology

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Superconducting radiofrequency (SRF) cavities are a key element in accelerator technology as they provide the accelerating field to circulating beams. High performance, state of the art cavities are made of bulk niobium with the drawback of the cost but also of the thermal instability because of the poor thermal conductivity of niobium.

Since 1980's CERN developed a technology of producing SRF cavities using copper bulk material coated with a niobium thin film. The usual coating technique was DC magnetron sputtering and has been used for all the LHC cavities. However these structures suffer from various problems. In particular, the degradation of the quality factor while increasing the accelerating field limits the use of such cavities at high accelerating gradients.

HiPIMS technology is thus seen as a good promise toward the improvement of thin films quality and density and thus toward cavities performance enhancement.

We will discuss on the effect of cavity biasing on the obtained Nb film morphology and properties. Plasma analysis carried out by mean of OES and retarding field energy analysis will also be presented to demonstrate the effect of a bias potential on the plasma parameters.

The difference between cavities processed using standard DCMS, unbiased and biased HiPIMS will also be outlined. We will finally propose routes for improvement specifically dedicated to our original geometry.

POSTER PRESENTATION

Manipulating HiPIMS deposition rates using magnetic field strengths

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The deposition rates in HiPIMS, conventional DC and pulsed-DC magnetron sputtering discharges have been compared for a variation in magnetic field strength. DC and pulsed-DC discharges show the expected behaviour that deposition rates fall with decreasing B (here by ~ 25 to 40 %), however the opposite trend is observed in HiPIMS with deposition rates rising by a factor of 2 over the same decrease in B.

From a simple phenomenological model of the sputtered particle fluxes, using the measured deposition rates as inputs, the combined probabilities of ionization, α, and back attraction, β, of the metal species in HiPIMS has been calculated. There is a clear fall in αβ (from ~ 0.9 to ~ 0.7) with decreasing B-field strengths, which we believe is due to a reduction in the size of the potential hill stopping ions reaching the substrate, so leading to higher deposition rates. This process is discussed further.

POSTER PRESENTATION

Influence of HiPIMS on the morphology of chromium nitride, (CrNx) films

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Chromium nitride films are frequently used as a adhesion interlayer in hydrogenated amorphous carbon (a-C:H) film systems. The morphology of these CrN_x interlayers has a direct influence on the surface quality of the a-C:H top layers. For example the corrosion protection properties of sputtered a-C:H films are limited by film defects which can originate from the interlayer.

Therefore, in this study the morphology of chromium nitride (CrN_x) films was optimized by high power impulse magnetron sputtering (HiPIMS). The deposition of the CrN_x films was accomplished by means of a Cemecon CC800 HiPIMS device. Polished 100Cr6 steel shims were used as substrates. The pulse duration and frequency of the HiPIMS cathodes as well as the bias voltage were changed systematically. The influence of these process parameters on the chemical composition, the microstructure and the mechanical properties were investigated by glow discharge optical emission spectroscopy (GDOES), atomic force microscopy, electron microscopy and microhardness measurements. Additionally, the corrosion behavior of selected HiPIMS-CrN_x/a-C:H film systems was tested by neutral salt spray test. With a decreasing duty cycle improved corrosion protection properties were observed due to the densification of the CrN_x film microstructure and a decrease of film defects.

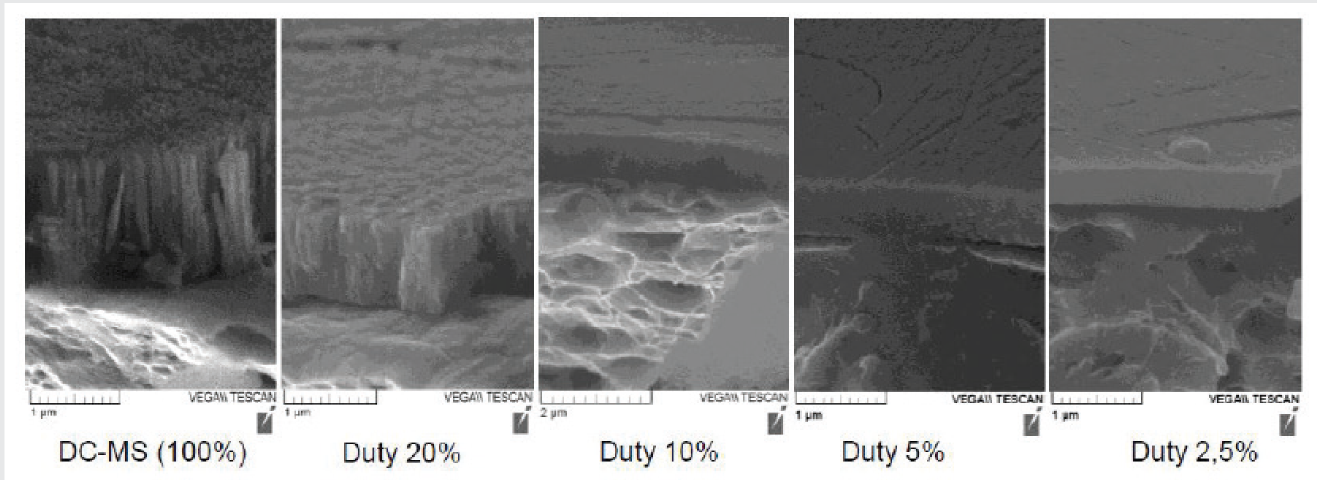

Figure: Influence of Duty-Cycle on morphology of CrN_x films

POSTER PRESENTATION

Deposition of TiSiN films by HIPIMS-DOMS: controlling the bombardment conditions by changing the Peak Power.

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Nanocomposite TiSiN films consist of nano-sized TiN crystallites surrounded by an amorphous Si – N matrix. Many works have shown that the hardness of nanostructured TiSiN films increases with increasing silicon content up to an optimal concentration. However, energetic ion bombardment of the growing film also influences the hardness and structure of TiSiN films. The main objective of the present work was to tailor the nanostructure of TiSiN films by using the highly ionized fluxes of sputtered material generated in a HIPIMS (High Power Impulse Magnetron) discharge. For this purpose TiSiN films were deposited by DOMS (Deep Oscillations Magnetron Sputtering) mode. The energetic ion bombardment of the growing films was controlled by changing the peak power.

The crystal structure of TiSiN films was analyzed by X-ray diffraction (XRD) with a parallel beam in both θ–2θ and GIXRD geometries. EDS and XPS were used to elucidate the chemical composition of the films and the nature of the chemical bonding, respectively. The microstructure of the films was characterized by SEM while their mechanical properties were measured by nanoindentation.

All the films deposited by DOMS have a nanocomposite microstructure consisting of two phases: f.c.c TiN and a-SiN. Although similar amounts of SiN were detected in the films, both the phase distribution and the properties of the f.c.c phase depend on the peak power. Two deposition regimes were identified. At low peak power (up to 44 kW) the growing film is bombarded with a high flux of low energy ionized sputtered species promoting the surface mobility of the ad-atoms and avoiding the atomic peening effect. At high peak power the energetic species impinging on the substrate are able to penetrate in the sub-surface of the growing film, resulting in an intense atomic peening effect which ultimately leads to secondary nucleation due to the high number of defects.

POSTER PRESENTATION

Mechanical Bending of the Indium Tin Oxide Films on Polyethylene Terephthalate Deposited by High Power Impulse Magnetron Sputtering

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Indium tin oxide (ITO) have been studied extensively because their high electrical conductivity and optical transmittance making them suitable for a variety of applications. ITO have been deposited on glass by high power impulse magnetron sputtering (HIPIMS) in the past demonstrating high electrical conductivity (>10^4 S / cm) and high optical transmittance (> 90 %). By considering the advantages of low temperature deposition capability, strong film adhesion and dense film brought about by using HIPIMS, this study evaluate the mechanical bending capability of the HIPIMS prepared ITO on polyethylene terephthalate (PET) so as to realize the feasibility of using HIPIMS for developing next-generation flexible electronics. Performance of the ITO PET commercial product obtained by using conventional DC sputtering is also compared.

The mechanical bending test was performed in compliance with the ASTM D1593 standard. Static and dynamic tests were performed separately. In the static bending test, the sample was bent to obtain a critical radius of curvature (Rc) where the electrical resistance abruptly increase. The durability for repeatedly bending cycles was tested at a specific bending curvature near Rc. Experimental results were discussed and explained.

Keywords: High power impulse magnetron sputtering (HIPIMS), Indium tin oxide (ITO), Flexible, Bending

POSTER PRESENTATION

TiO_2 thin film deposition by reactive multi-pulse HiPIMS

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High power impulse magnetron sputtering (HiPIMS) is a physical vapor deposition technique in which highly ionized deposition fluxes are generated by applying to the sputtering target short unipolar voltage pulses with power density values that exceeds by two order in magnitude the usual values in the conventional magnetron sputtering depositions. This technique does not only increase the deposition rate of the magnetron sputtering, but also improves the quality of the deposited films [1]. Recently, it has been shown that HiPIMS with pulses divided in sequences of very short micro-pulses, reffered here as multi-pulse HiPIMS, increases the ionization rate in the whole discharge volume, which leads to a better time-averaged conductivity of the plasma and to an improved ion transport towards substrate [2].

In this work, reactive multi-pulse HiPIMS of a pure Ti target (diameter of 50 mm) in Ar and O_2 mixture gas (mass flow rates of 20 and 0.2 sccm, respectively) at a total pressure of 10 mTorr is used to deposit of TiO_2 thin films. A single HiPIMS pulse of 15 µs was decomposed into a sequence of several shorter individual pulses (2 to 6 micropulses). The delay between the micropulses in a sequence was set to 50 µs. The average power injected into the discharge was kept constant (100 W) independently to the number of micropulses. For comparison, TiO_2 films were deposited by multi-pulse and single-pulse HiPIMS on glass substrates placed at a distance of 50 mm from the target.

The obtained experimental results show that the deposition rate in the multi-pulse HiPIMS increases with the number of micropulses, at the same average power it reaching four times the deposition rate of the single-pulse HiPIMS. This increase of the deposition rate is explained by limitation of the effect of reactive and metal ion back-attraction towards the target and by transition towards the metallic target sputtering mode. Thus, a larger amount of the sputtered material ionized in the HiPIMS micropulses are able to reach the surface of the growing film to produce an oxidized compound. Apart of the higher growth rate, the reactive multi-pulse HiPIMS produces stoichiometric and crystalline TiO_2 thin films with smooth surface and improved photo-catalytic activity.

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POSTER PRESENTATION

Corrosion and contact resistance characteristics of TaN_x films deposited by HPPMS on AISI 316L metallic bipolar plates in polymer electrolyte membrane fuel cells

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Tantalum nitride (TaN_x) thin films deposited at different N_2 / Ar gas ratios (0, 0.25, 0.625, 1) by high power pulsed magnetron sputtering (HPPMS) technique were evaluated for AISI 316L bipolar plate protection in polymer electrolyte membrane fuel cells (PEMFC).

The corrosion resistance of the coatings was measured by potentio-dynamic and potentiostatic polarization tests in 0.001 M H_2SO_4 solution (pH 3) at 80 º C, simulating operation conditions of a PEMFC. The study was performed just in cathodic environment where the conditions are harsher than in the anodic side. The potentiostatic polarization tests were carried out at 1.4 V_{she} during ex-situ testing (normal cathodic voltage operation in a PEMFC is 0.6 V_{she}). Such a high potential is applied in order to represent the typical peak voltage values occurring during real in-situ operation of a PEMFC. This fact is regularly ignored during ex-situ testing and leads to incorrect coating material performance assessment. All TaN_x films exhibit excellent chemical stability and lower corrosion currents than the uncoated AISI 316L substrate in the cathodic environment.

Interfacial contact resistance (ICR) was measured before and after potentio-dynamic and potentiostatic polarizations tests in the range of 40 – 400 Ncm^{-2}. The contact resistance of all TaNx coatings slightly increases after polarization tests, which indicates that the formation of oxide films cannot be completely prevented. The lowest ICR value is obtained for metallic Ta films deposited at N_2 / Ar gas ratio of 0. X-ray photoelectron spectroscopy (XPS) measurements were taken after polarization analysis to investigate the different oxide phase formation on the coating surfaces.

POSTER PRESENTATION

Oxidation resistance properties of TiAlSiN nanocomposite coatings on titanium alloy prepared by modulated pulsed power magnetron sputtering

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TiAlSiN coatings are developed to have good thermal stability at a high temperature over 800 °C. In this study, a series of TiAlSiN coatings were synthesized with different nitrogen/argon (N_2 / Ar) flow ratios by using modulated pulsed power magnetron sputtering (MPPMS) on titanium alloy. Cyclic oxidation behavior of TiAlSiN coatings with different N_2 / Ar flow ratios was investigated at 800 °C. TiAlSiN coatings exhibited excellent oxidation resistance properties at 800 °C for 50 hours over the uncoated titanium alloy. Before oxidation, the TiAlSiN coatings showed obvious x-ray amorphous characteristics. Transmission electron microscopy (TEM) observation showed a nanocomposite structure was formed in coatings, which have h-TiAlN nanocrystalline with a grain size of about 5 nm embedded in amorphous matrix. The silicon content in TiAlSiN coatings gradually increased from 6.1 at.% to 16.4 at.% with the decrease of titanium and aluminum content from 12.6 at.% to 10.3 at.% and 34.0 at.% to 19.9 at.%, when N_2 / Ar flow ratio gradually went up from 10 to 30%. TiAlSiN coatings prepared under 10% N_2 / Ar flow ratio possessed the highest aluminum and silicon content of about 40 at.%, while the others of about 36 at.%. All coatings showed similar hardness, modulus and residual stress of about 17 GPa, 225 GPa and -300 MPa. In the first 20 hours of cyclic oxidation, the TiAlSiN coatings sputtered at 10 % and 25 % N_2 / Ar flow ratios showed similar oxidation stability, the mass gains were only one tenth of the uncoated titanium alloy. After 50 hours, the oxidation rate of the coatings under 25 % N_2 / Ar flow ratio with high silicon content were obvious lower than the coating under 10 %.

POSTER PRESENTATION

Preparation and characterization of high purity Ti thin films by high power impulse magnetron sputtering deposition

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The increased ion-to-atom ratio in high power impulse magnetron sputtering (HiPIMS) allows directional deposition and film densification by the bombarding ions [1]. Recently, Andersson et al. showed HiPIMS gasless self-sputtering operation and proposed this method for the synthesis of ultraclean metal coatings through self-ion-assisted deposition [2]. In the present work we investigated Ti thin films prepared by direct current magnetron sputtering (dcMS) and HiPIMS with respect to their element composition, surface roughness, and microstructure. Ti films were deposited on Si / SiO_2 substrates at room temperature. The base pressure prior to the two hours depositions was 5 x 10^{-5} Pa. The film thicknesses were determined by profilometry after the deposition and are 800 nm and 200 nm for dcMS and HiPIMS respectively. It is shown that Ti thin films prepared by HiPIMS do not suffer from bulk contamination like dcMS films (Fig. 1). In particular, the impurity levels for O, N and C are below the detection limit (0.3 – 0.5 at.%) of elastic recoil detection analysis (ERDA) and the hydrogen content was measured to 0.5 at.% for the HiPIMS case. Compared to the dcMS films, we observed an element specific reduction of impurities by a factor 3 – 4 for N and H; and a factor of 20 for O. This suggests the presence of at least two sources of impurities. Unlike in [2], the HiPIMS self-sputtering regime was sustained in Ar gas. The high purity of Ti films can be partly explained by gas rarefaction and the cleaning effect of the bombarding ions. Moreover, densification effects presumably suppress post-deposition oxidation. The compositional effects are correlated with differences in the film microstructure revealed by SEM, XRD and TEM analysis. A more sensitive analytical method is needed to evaluate the actual impurity levels of O, N, and C in the deposited HiPIMS films.

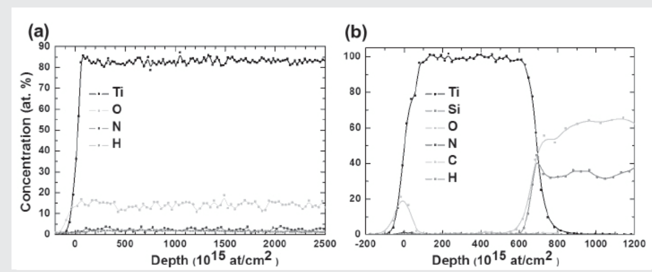

Figure 1. Depth profiled elemental compositions obtained by ERDA of (a) dcMS and (b) HiPIMS Ti thin films.

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Keywords: HiPIMS, self-sputtering, impurity level, Ti thin films

POSTER PRESENTATION

Influence of pulse off time on temporal evolution of sputtered species densities in HIPIMS discharge

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Developing of magnetron sputtering process is aiming mainly to increase ionization of sputtered particles, to enhance ion transport from magnetized region towards the substrate, to improve target yield and to enhance deposition rate. Sequences of successive short high-power pulses significantly increase the deposition rate compared to standard HiPIMS process keeping similar ionization efficiency of the sputtered species because the multi-pulse operation with short pulses limits the metal ion back-attraction [1]. Intensive gas rarefaction taking place during the HIPIMS pulse decreases the number of sputtered species and buffer gas atom collisions and influences the velocity distribution function of sputtered atoms in the direction parallel to the magnetron cathode as was experimentally shown in [2]. Consequently, the gas rarefaction should influence not only the deposition rate as it promote the non-collision transport of sputtered atoms from target to substrate but also it should decrease the resident time of the sputtered atom in the magnetized plasma and consequently also its probability to be ionized. As the buffer gas atoms needs relatively long time (at least several hundreds of µs as modelled by [3]) to diffuse back to the magnetized region from its perimeter, in the multi-pulse operation, the development of the subsequent pulse should be influenced by the preceding pulse. The local density of the buffer gas at the beginning of the pulse will be reduced by action the preceding pulses. A technique based on effective branching fractions was developed to determine the number densities of the sputtered titanium atoms and ions in their ground levels. The technique is based on fitting theoretically calculated branching fractions to experimentally measured fractions of the relative intensities of carefully selected lines of the measured species. Temporal evolution of the absolute number densities of sputtered species was measured for multi-pulse operation of the HIPIMS discharge as a function of the off time (0 - 1ms) in between the two successive pulses in the pulse packet. Influence of the gas rarefaction taking place during the pulse development manifests for the first pulse of the pulse sequence as a sudden saturation of the initially fast temporal evolution of sputtered atom densities followed after approx. 30 µs by sudden slow down of the initially fast temporal evolution of ionized sputtered species densities. The sudden change on the sputtered atom density evolutions is less distinctive for the second pulse of the pulse sequence but the density of both sputtered atoms and ion achieved at the end the second pulse is equal as the values accomplished at the end of the first pulse. Temporal evolution Ar line intensity suggests that the evolution of the argon gas density is not identical for the first and the second pulse in the pulse packed. The second pulse initiates at conditions of already reduces argon gas density in the magnetized region. Influence of the preceding pulse on succeeding pulse evolution was demonstrated even for the pulse to pulse off time of ~ 1 ms. The initial density of the sputtered atoms at the beginning of the second pulse was approx. half of the density obtained at the end of the preceding pulse showing slow diffusion of the sputtered atoms out from the magnetized region in between the successive pulses. However for off time of ~ 1 ms, the initial sputtered ionized species density drops almost to zero.

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POSTER PRESENTATION

Nanomechanical properties of nanocomposite coatings developed by HIPIMS and unbalanced sputtering

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Accurate characterization of the mechanical properties of nanocomposite coatings is particularly complicated, but it is crucial since these nanomaterials attract the interest of the scientific community and meet the needs of the protective coatings industry.

In this paper, we focus on the nanomechanical characterization of superhard nanocomposite diboride coatings using depth-sensing nanoindentation and atomic force microscopy (AFM). The coatings where developed using High Power Impulse Magnetron Sputtering (HIPIMS) and Closed-field Unbalanced Magnetron Sputtering (CFUBMS). The goal is to study in details the effect of the different growth technique to the nanomechanical performance of the nanostructured coatings.

The Nanoindentation testing was performed using two Berkovich (triangular-pyramid) type diamond indenters with different tip roundness (nominal tip roundness 20 nm and 50 nm), while the AFM was used to image the nanoindentation imprints and to study the difference in the deformation induced to the coatings by the two different Berkovich diamond tips.

The analysis of the nanoindentation load-displacement curves showed that the H values close to 52 GPa in the case of the CFUBMS coating. Finally, the mechanical properties were correlated with the thin film structure coming from the X-rays diffraction (XRD) and reflection (XRR) characterization.

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Hellenic National Strategic Reference Framework 2007 – 2013, contract no. 11ΣYN-5-1280, Project 'Nano-Hybrid', within the Program 'Competitiveness and Entrepreneurship'.

POSTER PRESENTATION

Comparative study DCMS vs HIPIMS depositions of TiO_2-flexible surfaces showing self-cleaning properties

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Compact uniform and adhesive thin films of TiO_2 deposited on flexible polymer have been prepared by DC-magnetron sputtering (DCMS) and high power impulse magnetron sputtering (HIPIMS). This study reports on TiO_2 sputtering on polyethylene (PE) showing effective and fast bacterial reduction kinetics [1] and methylene blue (MB) self-cleaning [2] properties under low intensity solar light. The dark / light bacterial reduction is reported as a function of the current intensities used for the sputtering processes.

More recently, the E. coli differential disinfection by HIPIMS and DCMS TiO_2 sputtered surfaces has been monitored and their OH° and O_2-generation performance has been compared [3]. The crystallographic "a" parameter and the nature of the surface-ions were quantitatively determined in both cases, to pinpoint the most relevant surface property leading to bacterial reduction. The atomic percentage concentration changes of the surface elements and the changes in the oxidation states of Ti were monitored during E. coli reduction by X-ray photoelectron spectroscopy (XPS).

These films PE-TiO_2 surfaces present potential practical applications for the disinfection since they preclude the formation of biofilms on PE, catheters and biomedical textiles.

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POSTER PRESENTATION

Comparison of TiN and Ti(C,N) coatings produced by HiPIMS and d.c. magnetron sputtering in an industrial coating facility

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HiPIMS differs significantly from the conventional d.c. magnetron sputtering, and together they cover a wide range of scenarios of deposition kinetics that are often critical for the characteristics of resulting coatings. In d.c. magnetron sputtering, particles sputtered from the target are mostly electrically neutral with low kinetic energies of only a few eV. In HiPIMS deposition, however, the particles ejected from the target crossing the highly densed plasma are largely ionized and, therefore, their kinetic energy can be purposefully adjusted before arriving at the deposition surface via an appropriate substrate bias. We therefore produced TiN and Ti(C,N) coatings using an industrial coating facility from both of the methods. The coating properties and microstructures were characterized and compared. The results will be discussed in this study and related to the strongly different deposition kinetics.

The coatings were deposited from a metallic Ti target in reactive mode with Ar-N_2 and Ar-N_2-CH_4 as working gas, respectively, under different target power, gas pressure and gas composition. Effects of thermal treatment were also examined on the deposited coatings. The chemical composition of coatings was analyzed by electron microprobe, the morphology by scanning electron microscopy (SEM), and the microstructure by X-ray diffraction (XRD). The coating hardness as well as reduced elastic modulus was characterized by nano-indentation.

POSTER PRESENTATION

Growth and mechanical properties of (Ti, Al) N films at inner wall of sub-millimeter scale small holes deposited by HIPIMS

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Demands for the surface modification at the inner wall of closed shaped sub-millimeter scale small holes are rapidly increasing. In order to enhance the film properties at inner wall of small holes, the authors focused on the great possibility of HIPIMS deposition, and have demonstrated its availability and practical advantages for microforming die applications [1]. For the further enhancement of the thin film properties at inner wall of sub-millimeter scale small holes, the present study investigates the relation between the mechanical properties and thin film growth inside the small holes in HIPIMS. As representative of the basic nitrides thin films, (Ti, Al) N film was deposited in the small hole structure, which has realized by clamping the comb-shaped stainless steel plate with two Si (1 0 0) substrates. To characterize the mechanical properties, nanoindentation tests were performed at the different depth position of holes structure under the several indentation forces. To investigate the factors of these obtained mechanical properties, the microstructure, local elementary compositions and its crystal structure at inner wall surface was analyzed in detail. In comparison with the thin film growth at the surface opposed to the target material, features of the growth at the inner wall surface of sub-millimeter scale holes were shown. Based on these results, the appropriate HIPIMS deposition conditions to obtain the uniform and high performance film coating were discussed in view of the application to microforming die.

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POSTER PRESENTATION

New Magnet Pack and Power Supply for High-Power Pulsed Magnetron Sputtering

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High Power Pulsed Magnetron Sputtering (HPPMS) is a type of magnetron sputtering technique where high peak power pulses reaching tens of kilowatts are applied to the sputter magnetron target keeping the average power equal to that of direct current magnetron discharges by using low duty cycles. Due to very high power densities, HPPMS discharge leads to high degree of ionization of the sputtered material. These ionized sputtered materials assist in film growth leading to more adhesive, dense, and smoother films. Therefore, HPPMS is considered an ideal candidate for the next generation magnetron sputtering systems. Two challenges exist to the broader adoption of HPPMS. The first challenge is the availability of HPPMS power supplies at output wattages better suited for circular planar magnetrons between 75 mm to 150 mm in diameter. The second challenge is managing an overall lower deposition rate due to “return effect” of the ionized sputter material [1].

To address the availability of appropriate power supplies the Kurt J Lesker Company in collaboration with Starfire Industries has developed a HPPMS supply that operates at output powers up to 2 kW with peak pulse currents of 200 A, pulse frequency of 30 Hz to 2.8 kHz, and pulse widths of 20 µs to 800 µs. The performance of the Impulse HPPMS will be demonstrated alongside Huettinger’s HiPIMS, MPP(zPulser), DC and pulsed DC power supplies. To address the “return effect” the Kurt J Lesker Company in collaboration with the researchers at the University of Illinois at Urbana Champaign has developed a magnetic field configuration for a 100 mm diameter Torus® that is optimized for HPPMS discharges. Magnetic pack design is critical as it helps in achieving full-face target erosion and higher deposition rate in HPPMS. A 100 mm diameter magnetron sputter gun typically has a conventional circular magnetic field configuration and suffers from low deposition rate in HPPMS discharges. To optimize the magnet field configuration in HPPMS for the 100 mm magnetron sputter gun, a spiral design from a previous 36 cm cathode design was scaled down and modified to fit into the 100 mm magnetron sputter gun. This new magnet pack with enhanced discharge parameters was developed by modifying the spiral magnet pack in COMSOL Multiphysics, which leads to higher deposition rate and better target utilization in HPPMS compared to the conventional magnet pack. The influence of the new magnet pack configuration on deposition rate, plasma parameters, and discharge stability with HPPMS (Huettinger’s HiPIMS), MPP(zPulser), Impulse, DC and pulsed DC power supplies are investigated.

6th International Conference on Fundamentals and Industrial Applications of HIPIMS – Braunschweig 2015 – Book of Abstracts 39

POSTER PRESENTATION

Structure and wear mechanism of novel CrAlBYCN/AlSiCN PVD coating deposited using a combined UBM and HIPIMS process in a reactive gas mix.

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A novel CrAlYBCN/AlSiCN coating has been devised for the improved wear resistance and high temperature stability of tools for cutting applications. The 2 µm thick nanoscale multilayer coating had a grain size of 11.2 nm, 11.1 nm and 6.6 nm as determined by XRD reflections from 111, 200 and 220 planes respectively. The as-deposited coating contained CrAlN and disordered carbon phases as detected by Raman spectroscopy. Hardness values were 3550 HK; the coating could be classed as superhard. Rockwell indentation showed class 0-1 adhesion and scratch test critical loads were 43 N on high speed steel substrates.

Isothermal annealing and thermogravimetric analysis of the coating indicated high temperature stability up to 900 ˚C and only a thin oxide layer supported by weak oxide Raman peaks and only 9 % oxygen inclusion at 800 ˚C observed by EDX. Heating to 1000 ˚C caused the coating to crack and delaminate from the stainless steel substrate and EDX indicated complete oxidation of the coating. In pin-on-disk tests with an Al_2O_3 ball counterpart at room temperature the wear track contained predominantly a CrAlN phase similar to the as-deposited coating. However the presence of faint CrN peaks indicated early signs of decomposition while Cr_2O_3 peaks indicated the onset of oxidation caused by localised flash temperatures estimated to exceed 1000 ˚C. Wear tracks produced at 200 ˚C contained a dominant Cr_2O_3 phase with little evidence of the original CrAlN structure, at this temperature the wear rate improved by an order of magnitude to 4 x 10^{-16} m^3 N^{-1} compared to room temperature. Raman of both temperature wear tracks showed clearly separated disordered and graphitic carbon peaks indicating that wear causes graphitisation of carbon in the coating which would improve wear resistance by acting as a solid lubricant.

POSTER PRESENTATION

Plasma Pretreatment of Tungsten Carbide and Steels by High Power Impulse Magnetron Sputtering

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Coated cutting tools are used for the majority of today's manufacturing operations. In a given cutting operation, the adhesion of the coating to the substrate is directly related to the lifetime of tools. Adhesion is commonly enhanced by the use of gaseous plasma to preclean the substrate and present a surface free of oxides for the growth of the coating. Metal plasmas are often more efficient due to the shallow implantation of metal into the substrate which enhances the wettability of the surface during nucleation of coatings of the same material.

The effects of metal ion implantation on the depth and chemistry of the interface and the microstructure of the surface are not sufficiently understood due to the relatively constrained parameter space available from conventional metal ion sources.

In this experiment tungsten carbide (WC), high speed steel and stainless steel were treated in the environment of a High Power Impulse Magnetron Sputtering plasma. The plasma chemistry was evaluated quantitatively by a combination of optical emission spectroscopy and plasma-sampling energy-resolved mass spectroscopy. Ion fluxes and deposition rates were measured simultaneously to obtain ion-to-neutral ratios. The measurements confirmed a strong rarefaction of the gas and indicated that rarefaction of the metal species may take place as well. Both single- and double-charged metal ions were detected. No significant delay between the gas and metal plasma was observed within a pulse.

The plasma diagnostics results were used as input to modelling calculations of penetration depth and chemistry near the substrate surface. Metal ions were found to penetrate approximately 4 nm into the WC substrate. The maximum implanted content of metal was found to increase as plasma became metal ion dominated and the metal ionisation degree increased.

Surface roughness of polished substrates increased due to the pretreatment as observed by atomic force microscopy, whereas as-received surfaces showed negligible differences. The etching removed preferentially smaller grains leaving behind a stronger substrate. Grain boundaries were also preferentially etched and the waviness factor was used to quantify the difference between samples. The etching rates corresponded to the ion flux to the substrate.

The mechanisms linking the plasma chemistry, surface chemistry and the adhesion of the coatings are discussed. Optimal parameters for improved adhesion are determined.

POSTER PRESENTATION

Time resolved ion energy distributions during HiPIMS of chromium: Transition from rotating spokes to a homogeneous torus at high plasma powers

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High power magnetron sputtering (HiPIMS) discharges generate ions with high kinetic energies in comparison to conventional dc magnetron sputtering. The origin of these high energetic ions is still an open issue. More recently, the formation of localized ionization zones (IZ) in the racetrack of a HIPIMS discharge, so called spokes, have been identified as a possible source of high energetic ions. This is explained by a local maximum of the electrical potential inside these localized IZ [1,2].

In this paper, ion energy mass spectrometry, probe experiments and plasma spectroscopy is performed at high temporal resolution. The data of a floating probe next to the target have been used to directly monitor the movement of the spokes in the E x B direction. Chromium is used as target material, because the plasma undergoes a sequence from homogeneous discharge, to stochastic spoke formation, to regular spoke pattern rotating in the E x B direction to a homogeneous plasma torus with increasing plasma power. This sequence is connected with the resulting ion energy distribution functions (IEDFs). Especially the transition from the regular spoke pattern to the homogeneous plasma torus at very high powers shows that the high energy part of the IEDF is not affected and only the low energy part is modified. Fig. 1 shows such a typical data set, where the transition from spoke to homogeneous regime is clearly visible in the probe data. At the same time, a characteristic change in the IEDF in Fig. 1b becomes visible.

Summarizing, one could consider the homogenous plasma torus at very high plasma powers a single ionization zone localized over the complete torus, which is formed by merging individual spokes with increasing power. Details and consequences of that model are discussed.

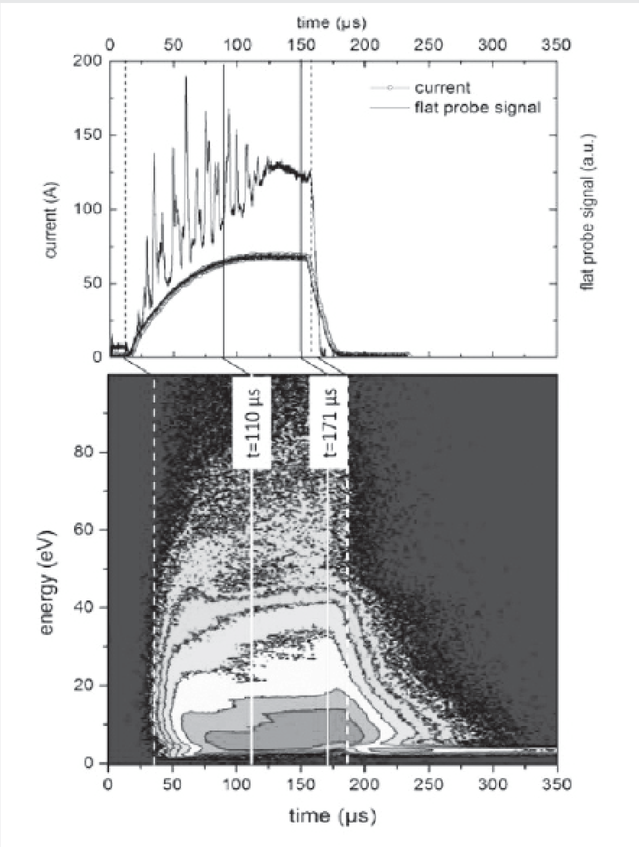

Fig. 1 (a) target current for 150 µs HiPIMS pulse at a 2'' chromium target and 0.5 Pa Ar (open circles). The target voltage is 700 V and the maximum current 70 A. The power density in the stationary phase is 2.45 kW /cm^2. Voltage signal at the flat probe (solid line); (b) time dependent energy distribution functions Cr^+ with a count rate plotted on a logarithmic scale. The dotted vertical lines denote the beginning and end of the pulse. The time scales are shifted with respect to each other due t the finite flight time between target and substrate.

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POSTER PRESENTATION

Laser scattering investigations of plasma turbulence in HiPIMS

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The nature of a variety of plasma instabilities is yet to be fully understood, particularly in plasma devices with crossed electric and magnetic eld congurations. The presence of such instabilities is generally driven by the density gradients, induced, for example, by dierences in charged particle velocities and eld gradients. Many of such instabilities are of interest because they govern plasma features such as particle transport and ionization. A full comprehension of the role they play in the physics of the discharge is required to validate plasma models and ultimately, improve the performance and predictive operation of complex magnetized sources.

Innovative experimental approaches can oer new ways to detect and characterize such instabilities. A highly-sensitive collective Thomson scattering diagnostic has been developed for the study of turbulence in low density plasma sources. This diagnostic, known as PRAXIS, measures electron density uctuations associated with the presence of instabilities at particular length scales. In the context of the Hall plasma thruster, this tool provided the rst experimental evidence (1) for the existence of an azimuthal electron cyclotron instability whose presence is associated with particle heating and higher electron currents across the thruster (2). This instability, driven by the fast azimuthal electron drift, has proven key in accounting for anomalous electron transport in regions of the plasma where collisional mechanisms are rare.

The PRAXIS collective scattering diagnostic has now been adapted for the rst time for the study of instabilities in a high power pulsed planar magnetron. Results from the

HiPIMS magnetron show that the electron cyclotron instability is indeed present in the pulsed magnetron plasma, at large amplitudes, and its role in transport cannot be neglected in favour of pure collisional mechanisms. The mode is detected at megahertz frequencies and at wavelengths on the order of the electron Larmor radius. Within a single pulse, the mode intensity is observed to oscillate at kilohertz frequencies, an observation which could be linked to the traversal of the diagnostic observation volume by plasma inhomogeneities such as rotating spokes. These results also provide experimental backing for the idea that this instability, driven purely by the fast electron azimuthal drift relative to the ions, is a common feature in all crossed eld sources featuring this type of drift. This work will discuss recent experimental results, their interpretations and implications.

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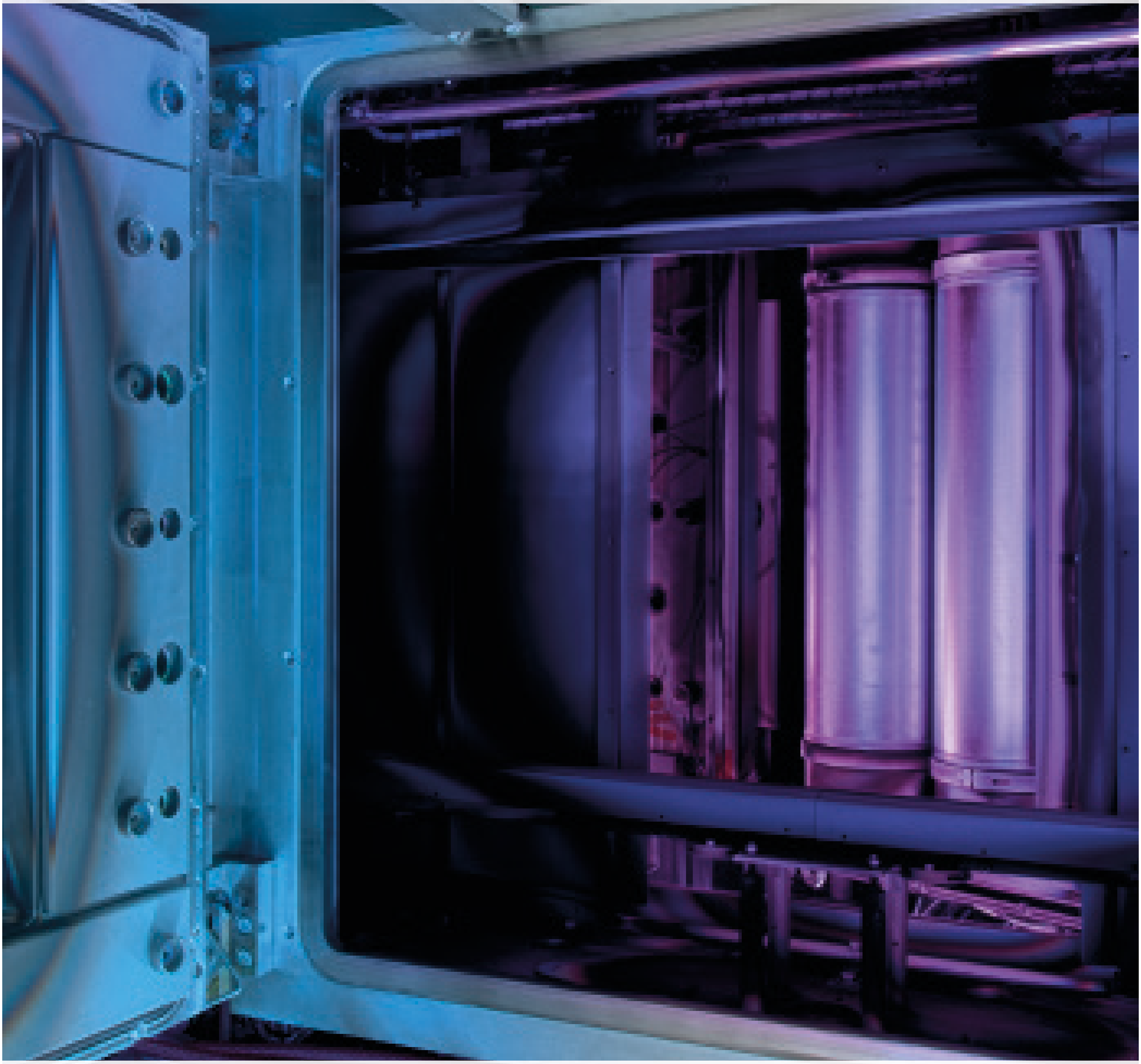

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