HIPIMS 2013

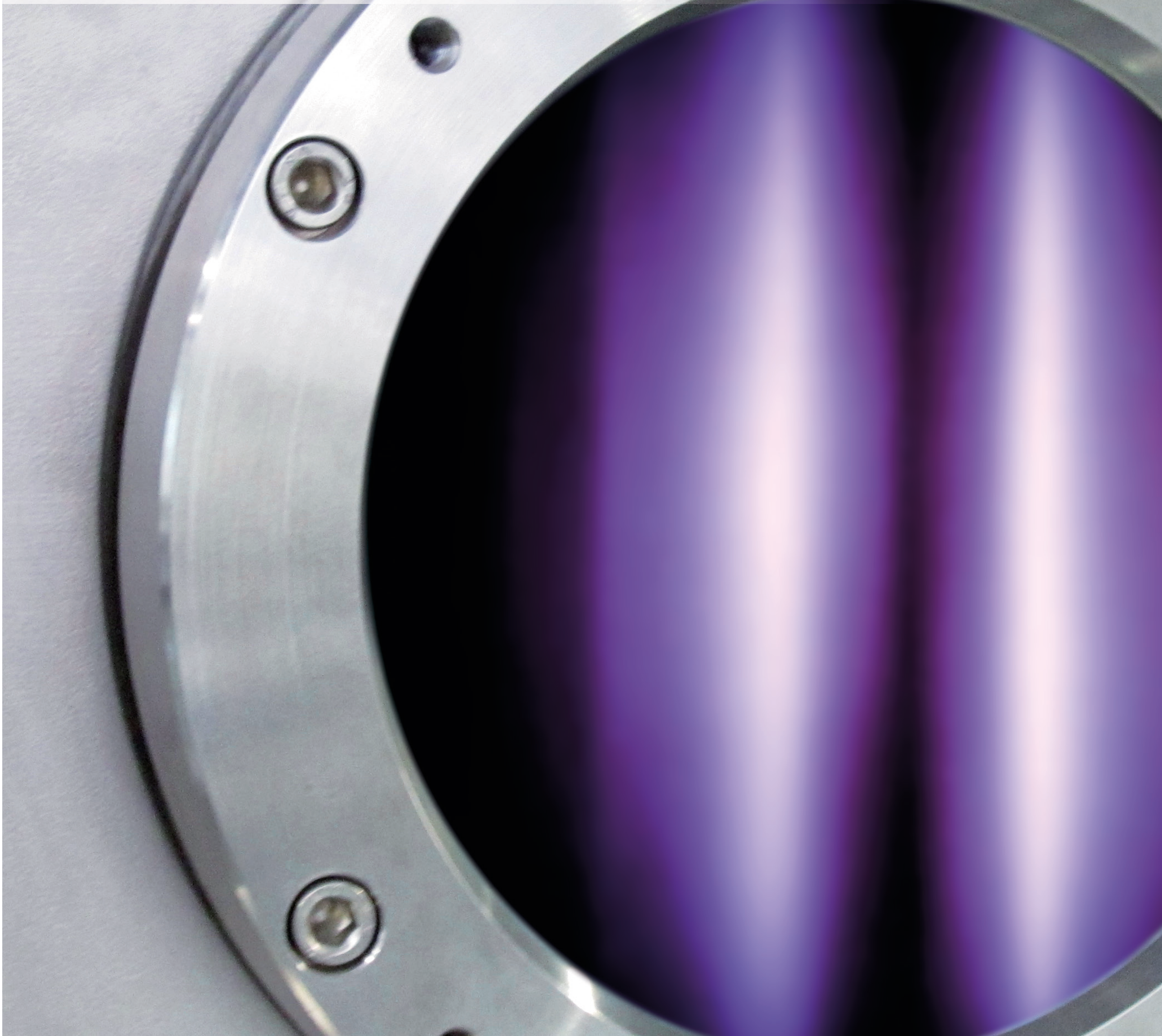
4th International Conference on HIPIMS

& Final Event COST Action MP0804

BOOK OF ABSTRACTS

4th International Conference on Fundamentals and Applications of HIPIMS

& Final Event COST Action MP0804



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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 approximately a decade of intense academic investigation and development we observe today a transfer of this new technology towards industrial processes.

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 120 delegates the impact of the new conference was underlined. The growing importance of HIPIMS technology was connected with a growth to more than 160 participants in the following two years in Braunschweig, DE and Sheffield, UK 2012 and 2013, respectively. The participants were made up of equal numbers from research and development (university and research institutes) and industry. Being a global conference representatives from 25 different countries from all continents attended.

The HIPIMS conference 2013 is linked with the Final Event of the COST Action MP0804 Highly Ionized Pulse Plasma Processes (www.hipp-cost.eu). COST (European Cooperation in Science and Technology) is one of the longest-running European frameworks supporting cooperation among scientists and researchers across Europe (www.cost.eu).

The COST Action MP0804 HIPP processes focuses on the fundamentals and the industrial implementation of highly ionized pulse plasmas, where HIPIMS is the most prominent and most mature technology, today. Within the Action representatives from 23 COST countries, one near neighbor country, and 4 NON-COST partners linked to create a unique forum to successfully accompany the transition of HIPP processes from fundamental science to applications.

The HIPIMS Conference 2013 and Final Event of COST MP0804 gathers world leading experts in the field of high power impulse magnetron sputtering (HIPIMS), on equal shares from academia and industry within more than 60 topical contributions. More than 70% of the contributions involve partners from the COST Action MP0804 and underline the close connection of the COST Action and the HIPIMS society, meeting annually at the HIPIMS conference.The talks from international experts covered a range from fundamental physics, experimental investigations, theoretically modeling to several applications and made the COST Action and this international conference on fundamentals and applications a success story to be continued in the following years.

Dr. Ralf Bandorf and Prof. A. Ehiasarian
(Conference Chairmen and Co-Chairman of HIPIMS 2013)

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ORAL PRESENTATION

HIPIMS as a tool to understand the time-domain and energetic bombardment effects on the nucleation and coalescence of thin metal films on amorphous substrates

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In the course of the past decade, HIPIMS has been established as an advanced ionized PVD method and extensive research has been performed to understand the fundamentals of HIPIMS process and plasmas and their implications for film growth[1-7].

We embark on the next era of HIPIMS by using its unique features as a science tool to improve our understanding of fundamental mechanisms during film formation. In the current contribution, we utilize the timedependent character of the HIPIMS process along with its high degree of ionization to study their effect on the nucleation and coalescence of thin metal films on amorphous substrates. Pulsed, ionized vapor fluxes, generated from high power impulse magnetron sputtering (HIPIMS) discharges, are employed Ag films on SiO_2 substrates. In situ plasma diagnostics and particle transport simulations show that vapor flux pulses with a width of ~200 µs, a frequency in the range 50 to 1.000 Hz and energies up to ~50 eV are provided to the growing film. Moreover, it is shown that the time-domain of the deposition flux and its energy can be controlled independently which allows for studying their effect on film growth separately. This is done by in situ monitoring of the film growth, by means of wafer curvature measurements and spectroscopic ellipsometry, determining the film thickness where a continuous film is formed (d_{coal}). The results reveal that dcoal decreases from ~210 to ~140 Å when either increasing the pulsing frequency for a constant pulse amplitude or the pulse amplitude of the material flux and the energy of the film forming species for a constant pulsing frequency. Estimations of adatom lifetimes and the coalescence times show that there are conditions at which these times are within the range of the modulation of the vapor flux. Thus, by changing the temporal profile of the vapor flux, nucleation and coalescence processes can be manipulated. It is suggested that, other than for elucidating the atomistic mechanisms that control pulsed growth processes, the interplay between the time scales for diffusion, coalescence and vapor flux pulsing can be used as a tool to determine characteristic surface diffusion and island coalescence parameters.

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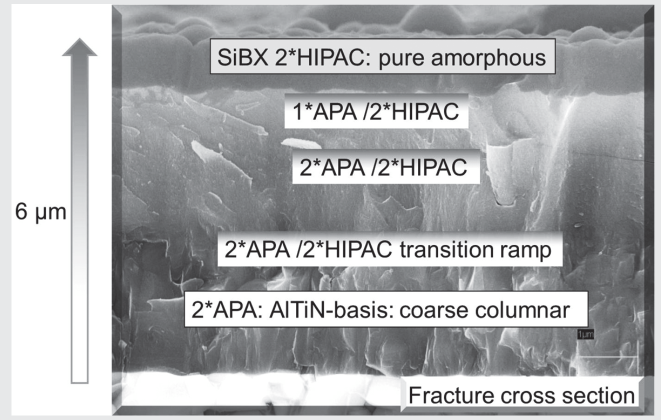
ORAL PRESENTATION

High ionization triple: An innovative PVD process for advanced coating architectures based on HIPIMS and Arc

J. Vetter, J. Mueller, T. Krienke, M. Schmidt-Mauer, G. Erkens

A new class of advanced PVD-coaters, the METAPLAS. DOMINO series , for dedicated coating applications comprise both improved vacuum arc evaporators (APA, Advanced Plasma Assisted) and high power impulse magnetron sputtering (HIPIMS) sources (HIPAC – High Ionized Plasma Assisted Coating). The ion cleaning is based on the (AEGD, Arc Enhanced Glow Discharge) process. This combination of the three highly ionized processes is named HI3 (High Ionization Triple). It's possible to run the processes in different modes, e.g. pure APA arc evaporation or pure HIPAC magnetron sputtering. However the combination of the two high ionized deposition processes to generate multilayer, nanomultilayers and nanocomposite layers opens 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, e.g. Si, SiC, WC, TiB_2 and others. Specific features of the PVD system equipped with APA arc evaporators and HIPAC magnetron sources will be shown. Selected results of hybrid coatings will be presented. Two advanced coating systems were successfully realized by the HI3 process: a coating architecture based on SiBX by HIPAC to increase the oxidation stability and, a coating type based on VXN by HIPAC to decrease the high temperature friction. Coatings were analysed by SEM, chemical analysis, nano-indentation hardness measurement and oxidation tests. Selected application results will be presented too. The enclosed figure shows the outstanding tailored coating architecture of SiBX coatings: from columnar to amorphous growth.



Example: Smooth gradient of the morphology
From coarse columnar growth to pure amorphous top layer

ORAL PRESENTATION

Advances in process technology and deposition equipment for HIPIMS coatings for cutting tools

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HIPIMS is characterised by short power pulses with an extremely short signal rise time.The design of the coating equipment need to take this characteristic into account with regard to feeding the electrical energy into the sputtering cathodes and finally into the plasma.

This paper will present recent results on the correlation of the hardware design of the machine and the coating process. Fundamental research about the efficiency of the pulse transfer and about methods to transmit an undistorted pulse shape and wave form into the process was done. The end user of a cutting tool sets its focus to the properties of the coating and, most important, to the machining characteristics of the film. Examples and field data will show how the most up-to-date HIPIMS coatings boost both productivity and quality. SEM images reveal a dense morphology of HIPIMS coatings. To this feature can be attributed that HIPIMS films combine high hardness and a relatively low Young's modulus indicating a high coating toughness in a way most favourable for metal cutting.

Super smooth coatings, free from any droplets, and low compressive stress are the most beneficial characteristics of sputter coatings for cutting tools. The effective bombardment of the growing film with highly ionized species further improves the surface of HIPIMS coatings.

ORAL PRESENTATION

TiN for forming applications

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TiN is one of the most used coatings for a number of different cutting and forming applications. The best TiN is typically produced by arc evaporation. However, for many precise forming applications, the abundance of droplets leads to high roughness at the surface of the coated part which necessitates after-treatment of the surface prior to the usage of the part. A large effort has been put in the last years to minimize the number and the size of the emitted droplets during the arc process. Nevertheless, extensive after-treatment of the coated parts is necessary in order to obtain the needed functionality. Recently, a lot of attention has been paid to high-density coating deposition by magnetron sputtering, especially highly charged techniques.

In the present work, TiN is produced by highly ionized magnetron sputtering where the charge Ti ions are abundant, reaching up to 90 % of the total Ti species, in the discharge, and the dissociation of the N_2 gas is increased. The coatings mechanical properties, including hardness, elastic modulus, surface roughness, and morphology are investigated both for parallel and angled depositions on the substrate. It is found that, compared to other conventional techniques, dense coatings are produced on all surfaces with much enhance coating homogeneity and properties.

ORAL PRESENTATION

Influence of Ion bombardment energy on the growth of CrN films by reactive magnetron sputtering and high power impulse magnetron sputtering

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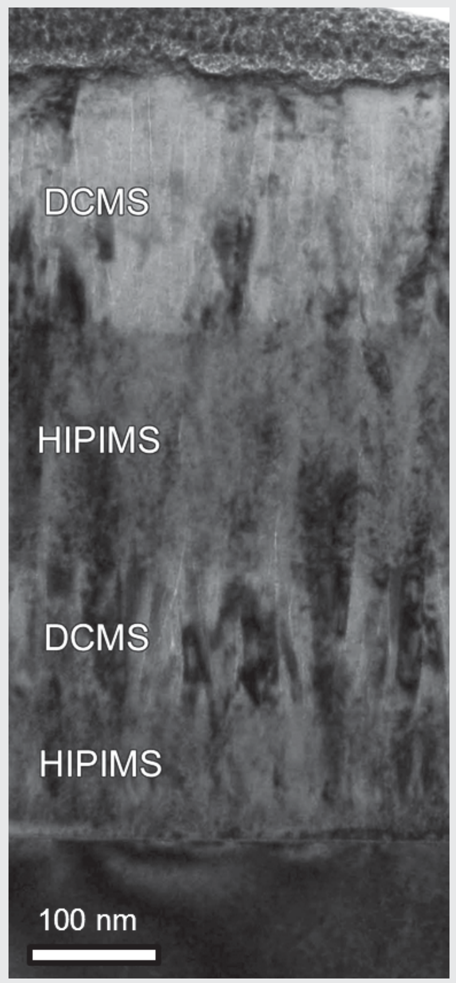
Production of transition metal nitrides with dense structures in industrially relevant conditions well below the homologous temperature requires assistance by ion bombarding flux with energy additional to that of a sputter process. In conventional direct current magnetron sputtering (DCMS) the metal species remain in a non-ionised state and have low energies. Thus, even at high ion-to-neutral ratios of 30, microstructures can be porous if ion energy is not augmented.

Recently, fully dense CrN films have been deposited at low temperature without substrate bias by high power impulse magnetron sputtering (HIPIMS) technology which ionizes the metal flux and dissociates nitrogen molecules within its plasma. It is not clear if the additional ionization provided by HIPIMS can outweigh a simple addition of energy by substrate biasing in DCMS. Therefore we compare floating and biased growth of CrN films by DCMS and HIPIMS technologies as shown in the cross sectional micrograph in the figure.

Mass spectroscopic analyses showed that HIPIMS deposition produces a factor of 10 higher flux of dissociated N^{1+} compared

to DCMS. A ratio of $N^{1+} : N_2^{1+} = 0.4$ in the HIPIMS plasma is factor 5 greater than in DCMS. The crystallinity of the DCMS layers improved with ion energy to a certain extent.However, a step change was observed when ionization degree increased in HIPIMS-process. The texture evolved from random towards (200) as energy and ionisation increased. (220) growth was eliminated altogether. In HIPIMS layers nucleation and competitive growth were resolved very quickly and the film structure was very dense. HIPIMS deposition on top of DCMS layers resulted in rapid closure of voids and establishing of a fully dense structure within 50 nm. Growth of DCMS layers on HIPIMS layers was unable to sustain textured growth because of the low ionization of the DCMS process. Results from DCMS-deposited films indicate that ion energy alone may be insufficient to promote a dense structure and a dominant (200) texture if $N^{1+} : N_2^{1+}$ ratio is too low. The HIPIMS results show that elevating the $N^{1+} : N_2^{1+}$ ratio to a moderate amount promotes the growth of (100) surfaces. Complementing this with moderate ion energy produces highly textured films with a fully dense structure.



ORAL PRESENTATION

Corrosion protection by HIPIMS+ deposited CrN-based coatings

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Because of the restrictions for use of hexavalent chrome, companies active in galvanic surface treatments are extensively using trivalent chrome. The corrosion protection of this surface treatment is not as good as for hexavalent chrome. For certain high-end applications, alternative treatments are required.

One advantage of HIPIMS technology is the possibility to make dense coatings with low defect density. In theory, this should result in improved corrosion resistance compared to arc and magnetron sputtered coatings. For the study, HIPIMS+ deposited CrN-based coatings have been deposited on steel substrates. The corrosion protection has been determined by salt-spray testing and will be discussed.

ORAL PRESENTATION

HIPIMS process with oscillatory voltage pulse shapes for directional sputtering applications

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HIPIMS / HPPMS is a fairly new technology that was developed in early 1990s. One of the important features of high power pulse magnetron sputtering is the ability to generate IMP (ionized metal plasma) in the most simple and cost effective way. The biggest applications of pulsed I-PVD technology could be directional sputtering processes to deposit Cu seed layers into different sized high aspect ratio features (trenches, vias and etc.) for integrated circuit fabrication.

In this presentation the experimental data such as the cross-sectional SEM images, voltage pulse shapes, pulse target power densities for directional sputtering of Ta and Cu in different sizes and aspect ratios trenches and vias will be presented. The experiments were performed with Zpulser Cyprium™ pulse plasma generator that can generate oscillatory voltage pulse shapes (oscillations frequency is about 20 – 60 kHz) and deliver up to 2 – 3 MW during the oscillation.

It was found that substrate ion current is a function of the voltage oscillations frequency and duration in the pulse. The substrates during all these experiments either had floating potential or were connected to the substrate bias power supply. DC power supply or RF power supply were used as a bias power supply. The RF bias is an important feature for the directional sputtering process for nonconductive substrates. The DC bias and RF voltage as a function of RF power at different level of peak power will be presented, as well as the experimental data about field, side and bottom coverage for trenches and vias.

ORAL PRESENTATION

Electrical characteristics of S3p™ HIPIMS discharge

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The new HIPIMS process S3p™ from Oerlikon Balzers allows optimizing high ionized sputter processes with regard to deposition rate, material flux ionization or hysteresis behavior in reactive sputtering. The full flexibility in pulse duration and pulse power density is a unique tool, opening up a wide range of possibilities in coating process development. Pulse length of 50 µs to 100 milliseconds and power density from 500 W/cm^2 to 2.000 W/cm^2 can be applied on a variety of sputter materials. A magnetron discharge is commonly described by the voltage – current characteristics. The characteristic is affected mostly by the gas rarefaction effect, current density, secondary electrons, compound formation or the target material.

The present work will give examples of Voltage-current density characteristics by S3p™ HIPIMS discharge, in comparison with the work of other research groups. Influence of pulse duration, gas pressure and reactive gas will be discussed in detail.

ORAL PRESENTATION

New development of HIPIMS power supply with best in class technology and new features

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Our HIPIMS power supply developed years ago, is the first device, which enables sputtering materials with HIPIMS technology on an industrial scale. Based on the long experience in HIPIMS research as well as in industrial applications, like tool coating, medical, microelectronics, optical, photovoltaic, TruPlasma Highpulse 4000 New HIPIMS power supply has been developed. In the paper new features enabling improving process parameters and quality of deposited layers are presented.

Especially highlights like: arc management with a various arc detection conditions, optimizing arc management, arc quenching circuit (CLC) minimizing arc energy, full water cooled platform providing high level of compactness, advanced control and monitoring circuits facilitating optimization of process parameters, are discussed.

Finally application of new features and their influence on parameters of HIPIMS processes is shown.

ORAL PRESENTATION

Deposition rates and oxygen negative ion energy distributions during reactive HIPIMS of titanium in Ne /O_2, Ar/O_2, Kr/O_2 and Xe/O_2 gas mixtures

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Reactive sputter deposition, particularly in the presence of electronegative gases such as oxygen, often leads to the formation of high energy negative ions. These energetic negative ions are formed at, or close to, the target surface and can be detected at the substrate with energies close to the applied target potential. The impingement of energetic negative ions onto the substrate has been shown to have detrimental effects on the structural[1] and electrical[2] properties of the growing film and in some applications it is desirable to mitigate this bombardment while maintaining a high deposition rate.

In this contribution, the deposition rates of reactive HIPIMS of Ti in a gas mixture of oxygen and different rare gases (Ne, Ar, Kr and Xe) have been measured using a quartz-crystal microbalance (QCM). The QCM was positioned above the racetrack directly facing the target surface at different axial positions (50 and 100 mm above the target surface). The HIPIMS discharge was operated with a pulse on-time t_{on} = 100 µs, a pulse frequency f_p = 100 Hz and at a constant average discharge power P_{avg} = 100 W. The partial pressure of oxygen was maintained constant at 0.2 p_t where pt is the total pressure; p_t was varied from 0.4 to 1.2 Pa. By means of energy-resolved mass spectrometry, the energy distribution of oxygen negative ions (O-) was also measured at the same positions and using the same HIPIMS discharge parameters.

It was found that for reactive HIPIMS using oxygen mixed with heavier rare gases (i.e. Kr and Xe), the measured intensities of high energy O^- ions was reduced significantly when compared with those in lighter gas mixtures (i.e. Ne/O_2 and Ar/O_2). Furthermore, the normalized static deposition rates of reactive HIPIMS in Kr/O_2 and Xe/O_2 were found to be higher than those measured when using Ne/O_2 or Ar/O_2 gas mixtures at low pressures. It is proposed that this reduction in the intensity of high energy negative ions is due to a combination of a larger collisional cross-section during the gas transport phase as well as a lower ion-induced secondary electron emission coefficient for the Kr/O_2 and Xe/O_2 gas mixtures, which has previously been shown to be correlated with high energy negative (O^-) ion emission during reactive magnetron sputtering.[3]

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ORAL PRESENTATION

Plasma characterisation of the HIPIMS instabilities by energy resolved mass spectrometry

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HIPIMS plasma discharges are characterised by high plasma densities and high ionised particle fluxes. These are the key plasma parameters for the growing film quality in the ion-assisted deposition process and characterisation of the plasma is essential to understand and improve the deposition process.

Recently instabilities have been observed in HIPIMS [1-2] that show the plasma formed is highly complex and inhomogeneous. The instabilities are well – defined bright and dark regions of plasma arranged periodically along the full length of the racetrack of a circular HIPIMS magnetron [2]. The instabilities result in intermittent enhanced particle transport away from the cathode which may be of significant technological importance [2]. These add complexity to the understanding of the spatial and temporal dynamics of the particle fluxes (ions, neutrals and radicals) and it is of great importance to investigate this phenomenon in detail to build a better understanding of the HIPIMS discharge. In particular, we have seen that the instabilities behave differently for different values of the peak current value, changing from a stochastic situation to a periodic rotation situation [2].

The present work reports the results of an investigation with QMS for different values of the peak current. The energy distributions of ions and neutral-metal species, »energetic neutrals«, from a target surface have been measured using a Hiden EQP energy/mass resolved spectrometer. The behaviour of the HIPIMS discharge was investigated for many different voltage conditions ranging from 400 to 900 V in a pure Ar atmosphere for a copper and chromium target. The integration of the ion energy distribution function (IEDF from the neutral-metal »energetic neutrals» gave a relative neutral flux of both targets at different current conditions. The results obtained are shown in Figure 1 and it was found that the represented values as a function of the applied current to the target follow the trend of the Voltage-Current characteristics.

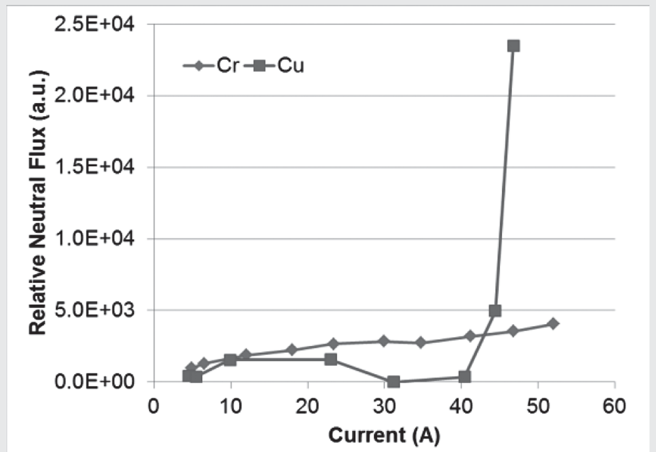

Figure 1: Relative neutral flux of Cu and Cr target at different current target values

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ORAL PRESENTATION

A novel, deposition-tolerant, Langmuir probe suitable for plasma parameter measurement in HIPIMS discharges

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Conventional Langmuir probes are difficult to operate effectively in deposition plasma processes, such as HIPIMS, especially when insulating layers are deposited. A novel radio-frequency (rf) biased Langmuir probe (Plato) has been developed to overcome the issues that arise when insulating layers are present.

Traditional Langmuir probes are cleaned through dc bias induced electron bombardment of the probe tip. This method is not possible in the presence of an insulating layer. Time resolved plasma measurements, through the HIPIMS pulse, using the standard 'boxcar' technique is difficult since the plasma can change from pulse to pulse. Changes in the work function of the standard Langmuir probe tip material due to contamination can also become problematic over time. Our novel rf biased probe provides a solution which overcomes most of these issues. Cleaning is not required since the rf bias will penetrate any layers deposited. A probe scan is performed in one rf period which takes place on the order of a micro-second. Therefore, the plasma parameters in individual HIPIMS pulses can be resolved with micro-second time resolution. The »boxcar« method is therefore not needed. Problems due to changes in the work function are avoided since the rf bias is not sensitive to dc fluctuations.

The Plato probe consists of an aluminium disk to which a rf bias is applied since the dc bias sweep can not penetrate the insulating layers. Experiments are compared for insulated probes (coated with a hard-anodized layer and conducting probes to validate the technique.

This technique relies on accurate measurement of the rf current and voltage waveforms at the electrode surface. The measured radio-frequency current consists of particle fluxes (real) as well as displacement current (imaginary) – due to the charge stored in the capacitive sheath that necessarily forms in front of the planar electrode. The radio-frequency voltage applied to the capacitive sheath and real time dependant particle current are determined to enable the computation of the plasma parameters.

A novel algorithm is described which allows the real particle current to be derived from the measured waveforms. Plotting the real current versus the sheath voltage yields the real current-voltage (IV) characteristic similar to that of a standard Langmuir probe. Analysis of this characteristic gives the plasma parameters in the usual way.

Plasma parameters determined with the radio-frequency biased deposition-tolerant probe (with and without the insulating layer) are shown to be in excellent agreement with independent, standard Langmuir probe measurements taken in non reactive plasmas. This method continues to operate well when additional layers are deposited by plasma process.

ORAL PRESENTATION

Fe_2O_3 thin films for water splitting application prepared by high power pulsed magnetron and pulsed hollow cathode systems

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Fe_2O_3 semiconductor material was recently found very promising as a photoanode for water splitting application. Semiconductor oxide thin films of Fe_2O_3 were deposited by reactive sputtering in a high power pulse magnetron and in a pulsed hollow cathode plasma jet sputtering system without external heating of the substrate. It was possible to prepare by both methods nanocrystalline or polycrystalline thin films of hematite Fe_2O_3. HIPIMS conditions were capable to deposit smooth, dense nanocrystalline hematite. Application of different magnitudes of duty cycle was tested in the range 1 % – 50 %. Quite different preferred orientation of crystallites relative to the substrate surface was observed in dependence on the duty cycle. This texture was apparent at as deposited films and also at thin films annealed after deposition on 600 °C. Hematite Fe_2O_3 thin films were deposited also by the low pressure DC pulsed hollow cathode system. The metallic hollow cathode with internal diameter 5 mm and length 30 mm was sputtered in argon plasma flow and reactive gas oxygen was supplied directly to the reactor. The hollow cathode discharge was generated by the DC pulsed power supply working in high power pulsed mode. The maximum attained pulsed current density in our hollow cathode discharge was approximately ≈ 3 A/cm^2. The main advantage of this system was the high deposition rate which was nearly independent on the amount of used oxygen in the plasma. Fe_2O_3 hematite thin films were deposited with this system. Surface roughness of films was much higher for the pulsed hollow cathode system than for pulsed magnetron system. Photoconductivity and photoelectrochemistry under 1AM illumination was measured on deposited on Fe_2O_3 thin films. IPCE (induced photocurrent efficiency) was obtained for these samples.

POSTER PRESENTATION

Can HIPIMS Cr_xN serve as alternative for hard chromium?

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Electroplated hard chromium is a common surface finishing approach in many engineering industries due to its high hardness and excellent corrosion resistance. However, environmental and health regulations on chromates drive the exploration of alternative coating methods, among which physical vapor deposition looks very promising.

This study aims to evaluate PVD CrxN as a viable replacement for hard chromium. A way to achieve an increased performance of PVD coatings in terms of wear and corrosion resistance is the use of High Power Impulse Magnetron Sputtering. The HIPIMS technology is believed to allow for denser coatings with superior mechanical, tribological and wear resistant properties compared to DC-MS.

Cr_xN coatings are deposited by means of DC-MS, HIPIMS and combined DC-MS/HIPIMS. Mechanical (Calo test, Nanoindentation test, Scratch test, 3D Profilometry, Taber Abrasion test) and tribological (Pin-on-disc test) properties and corrosion resistance (Salt spray test) of the different coatings are studied and compared to electroplated hard chromium.

POSTER PRESENTATION

Correlation between the Rockwell indentation test and the progressive load scratch test for assessment of coating adhesion

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In recent years it has become current practice to employ a wide range of test methods to assess the adhesive properties of thin films and coatings, ranging from the simple tape-peel test to the more advanced pull-off and 3-point bending tests. Many such methods have high operator subjectivity, high data variability and in many cases are unsuitable for industrial Quality Control (QC) use where assessment is done regularly and often by several operators. The Rockwell indentation test (ISO 26443 and VDI 3198) has become common for evaluating the adhesion of hard coatings deposited on various substrates, but the applied load (1472 N) is often too high causing the 200 µm radius diamond indenter to plunge too deeply into the substrate making it difficult to classify the resultant coating damage around the edges. The progressive load scratch test (ASTM C1624, ISO EN 1071-3, ISO 20502) is now widely used to characterize the adhesion of thin hard coatings with high repeatability. However, a direct correlation between these two methods has never been attempted.

It has been proposed that a smaller radius diamond indenter (possibly 50 µm radius) could be employed with the Rockwell indentation test, using a lower applied load, in order to concentrate the maximum stress nearer to the sample surface causing more focused failure of the coating-substrate interface. The objective of this study is to fabricate samples representative of the 6 failure categories of the standard Rockwell test, to then evaluate the same coatings with lower load and smaller indenter radius, and thirdly to test the same samples with the progressive load scratch test. It is hoped that this could lead to better correlation of data obtained with all such methods and aid industry in better QC control of their coatings.

POSTER PRESENTATION

Target implantation and re-deposition processes during high power impulse magnetron sputtering of aluminium

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The processes of argon retention by the target and re-deposition of target material were investigated by X-ray photoelectron spectroscopy (XPS) as function of radial position for different plasma conditions in high power impulse magnetron sputtering (HIPIMS) of aluminium targets. Significant differences in Ar radial concentration profiles were observed for different discharge conditions. Since the presence of a permanent reservoir of gas within the target can influence the characteristics of the sputtered material flux (in terms of angular and energy distribution, for example), it is important to understand the plasma conditions for which Ar retention by the target takes place and in what magnitude. Inside the racetrack area, Ar ion flux-dominated implantation is compensated by radiation-enhanced diffusion loss terms. Outside the racetrack, the role of ion implantation is diminished, and Ar retention by the target may stem from a balance between gettering by redeposited Al and ion-induced Ar desorption.

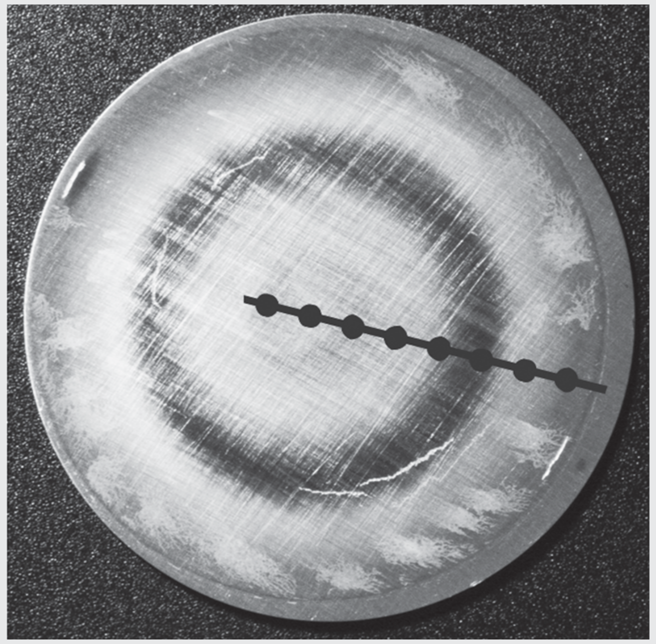

Photo of a 2 inch Al magnetron target exposed to a HIPIMS discharge for 3 minutes. Superposed are the points where XPS characterization was performed, in order to obtain the radial concentration profile of Ar gas retained by the target

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POSTER PRESENTATION

Mechanical properties of TiSiN coatings processed by HIPIMS-pulsed DC hybrid process

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Superhard TiSiN coatings attracts great interested since they exhibit high hardness > 50 GPa[1]. The reason of the enhanced properties is attributed to the possibility of tailoring the TiN size – in the nanometer range – by the content of the amorphous SiNx located at grain boundaries[1, 2].

In addition, it presents an improvement in terms of oxidation resistance if compared with TiN which allows the application on high speed cutting tools[3]. The reason is attributed to the segregation of SiNx on the grain boundaries which acts as a shield, able to protect the corrosion-prone nitride grains from an aggressive oxidizing atmosphere[4]. As a matter of fact, during oxidation the migration of Si to the surface accompanied by the formation of SiO_2 barrier lead to improve the refractory character[5].

Several works were devoted to TiSiN nanocomposites thin films processed by chemical and physical vapour deposition processes. Among the physical vapour deposition processes, several studies centered the attention on the conventional DC or RF sputtering and/or arc evaporation. However, a lack of information regarding the deposition by using High Power Impulse Magnetron Sputtering (HIPIMS) was found.

In this study, samples were fabricated by co-sputtering of Ti and Si targets in order to investigate the effect of nitrogen flow rate and bias voltage on the deposition of TiN by HIPIMS. Hence, the effect of silicon content introduced into the coating by co-sputtering the Si target using conventional pulsed-DC was studied owing the comparison of the final properties – i.e. microstructural, morphological, mechanical and tribological.

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POSTER PRESENTATION

Gyrokinetic description of technical plasmas

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Many technical plasma processes, like magnetically enhanced reactive ion etching (MERIE), plasma ion assisted deposition (PIAD), and of course conventional and high-power impulse magnetron sputtering (dcMS/HIPIMS) employ (partially) magnetized high density plasmas at relatively low pressures. (Typical values are $p \approx 0.01 - 1$ Pa, $B \approx 100$ mT, $n_e \approx 10^{16} - 10^{19}\ m^{-3}$.) These plasmas are, at least in their active regions, characterized by a peculiar ordering of the dynamic length and corresponding time scales. Using λ_D, s, rL, L, λ, and λ^* to denote the Debye length, the sheath thickness, the gyration radius, the system length, and the elastic and inelastic electron mean free paths, respectively, the length scales obey

$$\lambda_D << s << r_L << L \lesssim \lambda << \lambda^*. \quad ^1$$

It is well-known that discharges of this regime are hard to analyze. Conventional fluid models do not apply, and kinetic methods must be used. Numerical simulation using an explicit particle-in-cell scheme (PIC), however, is extremely costly: Its effort number $N = \omega_{pe} T\ (L / \lambda_D)^3$ (timesteps × gridpoints at 3D) may exceed 10^{18}.

Our contribution investigates an alternative kinetic description known as gyrokinetics. This approach – actually not one theory but rather a family of theories – very was successfully used in the field of high temperature plasmas[1,2].
It is based on the insight that the fast gyro motion of the charged particles in magnetized plasmas can be mathematically separated from the slower drift motion and »integrated out«, leaving only an effective dynamics which describes the longer time and larger length scales.

Our manuscript will present a transfer of this idea to technical plasmas. It will in particular focus on the questions how technical (i.e., low temperature) plasmas differ from their high temperature cousins, and how those differences – different field topology, magnetization of only the electrons, presence of material walls, presence of neutral gas, etc. – are reflected in the mathematical description.

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POSTER PRESENTATION

Influence of the Y-doping on the oxidation and mechanical properties of AlCrN-based coatings

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The study AlCrN hard layers for high speed machining applications increased during the last years, as commercially available coatings present high abrasion and oxidation resistance over 800 °C, thus increasing the tool lifetimes. Recently, the addition of alloying elements such as yttrium are believed to be suitable for enhancing the oxidation resistance at high temperatures, as reviewed by several authors[1].

In this study, AlCrN coatings have been deposited by high-power impulse magnetron sputtering (HIPIMS). This innovative technique has been chosen due to its enhanced final properties in order to improve the current coatings usually processed by cathodic arc evaporation technique.

The results were discussed taking into account the role played by the yttrium content in AlCrN coatings. For this purpose, thermogravimetric measurements were performed in order to evaluate the role of the yttrium doping if compared with the non-doped material.

It is believed that surface and bulk oxides are sufficiently plastic to sustain abrasive wear at high temperatures without spalling effects[2]. Furthermore, they are supposed to increase the wear resistance as they reduce the friction coefficients due to the oxides' formation which act as protective layer[3].

Microstructures and morphology of the deposited coatings, before and after tests, have been investigated by means of X-ray diffraction and scanning electron microscopy (SEM). Nonetheless, the coatings' oxidation as a function of temperature and time was quantified by glow discharge optical emission spectroscopy (GD-OES).

Finally, samples were submitted to mechanical (nanoindentation) and tribological characterization with the aim of correlating the final microstructures to the mechanical behavior.

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POSTER PRESENTATION

Study of wear mechanism of chromium doped DLC coating by Raman spectroscopy in boundary lubrication condition

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The aim of this work is to study the wear mechanism of chromium doped DLC coating (Cr-C) in dry sliding and boundary lubrication condition. The amorphous Cr-C coating was deposited by High Power Impulse Magnetron Sputtering (HIPIMS) process on hardened (62 HRC) high speed steel substrate. The friction coefficient of the coating was measured by pin-on-disc test under dry and boundary lubricating conditions. Highly viscous commercially available engine oil (Mobil1 10W-60) was used as lubricant. The Raman spectroscopy and X-Ray Diffraction were used to characterise the wear products to understand the wear mechanism.

It was found that compared to dry sliding condition, under lubricating condition the friction coefficient of the coating reduces from $\mu = 0.21$ to $\mu = 0.18$ (~ 14.3 %). Raman spectrum was collected from different positions of the wear track after dry and oil lubricated sliding to investigate the wear products and in turn to understand the wear mechanism. Raman spectrum indicates that in dry sliding condition chromium and iron oxide peaks were present, which was expected due to the operation of the oxidative wear mechanism. However, no oxide wear particles were detected when the sliding took place in oil lubricating condition. Unlike dry sliding condition, the wear product found in the wear track was a mixture of chromium carbide, chromium chloride and chromium sulphide. The XRD anlyses confirmed the presence of these compounds. The most intensive peak in the XRD pattern was assigned to chromium sulphide whereas comparatively lower intensity peaks were recorded for chromium carbide and chromium chloride. Based on these findings, the wear meachnism can be described in the following way. In boundary lubrication condition, the carbon, chlorine and sulphur compounds from the oil react with the metal dopant of the coating (Cr) due to high flash tempeartures (~ 800 ˚C) at the asperity contacts. The reaction product is mainly chromium sulphide and comparatively less amount of chromium chloride and chromium carbide. These compounds are adhered to the wear track by forming a thin layer. Among these compounds, chromium chloride has a graphite-like layer-by-layer (low shear strength atomic planes) structure, which acts as a solid lubricant.

Therefore it could be speculated that in boundary lubrication condition, the reduction in friction coefficient is achieved due to the formation of solid lubricants such as chromium chloride via tribochemical reaction mechanism.

POSTER PRESENTATION

Correlation between mass-spectrometer measurements and thin film characteristics using HIPIMS discharges

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In this work, chromium and chromium nitride thin films were deposited using HIPIMS technology [1]. This IPVD technique provides a lot of interest and is used in the coating community since last decade. Inject the power on a short time allows us to apply a very high power density on the target. Thus, we achieve a high ionization of the metal vapor and by this way, we can enhance the adhesion, the crystallinity and the hardness, controlling the ion flux toward the substrate. That's why we attempt here to establish the link between plasma and coating properties.
Numerous experimental parameters can be used to drive the ion flux i.e. the thin film properties. Among them, duty cycle, pulse width, are studied to better understand their real effect. One way to estimate the number and the energy of the ion species consists in mass-spectro-metry measurements. Here we use for the plasma diagnostic a mass-spectrometer resolved in energy from Hiden (EQP 1000). We choose to associate it with a multichannel analyzer (Ortec) to obtain a time resolution of 2 µs. Thereby, the delay of arriving species, decay time in the post discharge can be analyzed precisely.
A correlation between the in situ diagnostics and material analysis will be made. Classical techniques such as XRD, SEM, TEM and XPS were carried out for the material analysis.

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POSTER PRESENTATION

The study of titanium nitride films deposited using a hybrid system combining cathodic arc deposition and high power impulse magnetron sputtering

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It was known that cathodic arc deposition (CAD) has been widely used in industry for high quality thin film coatings. An extremely high current density (~1012 A/m^2) was created to evaporate and ionize the target material rapidly. However, the CAD also produces macro particles or droplets along with the deposition process leading to degrade the film properties. Magnetic filter with different design was therefore adopted to reduce the macro particles or droplets. However, the macro particles or droplets still can't be fully eliminated. Lately, a newly developed PVD process known as high power magnetron sputtering (HIPIMS) was found to have the capability of yielding highly ionized flux of both gas and sputtered materials by applying a high power in short pulses to the target. High plasma density in the order of 1017 to 1019 m^{-3}, which is three orders of magnitude higher than the conventional dc magnetron sputter (dcMS), can therefore be achieved from the large fluxes of energetic ions in HIPIMS technique. As a result, a smoother and denser thin film with better adhesion to the substrate can be obtained, leading to enhanced mechanical, electrical, and optical properties. However, it was also found the deposition rate of the HIPIMS process was much slower than conventional dcMS. Therefore, a hybrid deposition system combining CAD and HIPIMS was studied in this paper. Furthermore, titanium nitride (TiN) film was deposited in such hybrid system to investigate their microstructure and the mechanical properties such as surface roughness, hardness and friction coefficient. A rectangular and two round Ti targets were used in HIPIMS and CAD techniques, respectively. A mixture gas of argon and nitrogen was introduced to reach the deposition pressure of 2×10^{-2} torr. Besides, the alternating and simultaneous process of these two deposition techniques was also investigated in this paper.

POSTER PRESENTATION

Time-resolved optical emission spectroscopy of a vanadium HIPIMS plasma

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HIPIMS is a sputtering technique that enables ionised deposition in the absence of macroparticles and is therefore attractive for applications seeking to improve film quality through ion bombardment. Due to the pulsed mode operation the plasma properties vary strongly with time. Optimising the production of ions and the charge state distribution requires knowledge of how these properties evolve through the pulse and how they are affected by changes in operating conditions. In this study, we use optical emission spectroscopy to examine the evolution of the charge states present as a function of time and pulse voltage.
As described in a previous publication [1], highly spectrally and temporally resolved Optical Emission Spectroscopy (OES) using a large number of spectral lines can be used to investigate the underlying plasma processes and relate them to the system parameters. In [1] it was shown that the optimum energy efficiency of production of titanium ions in HIPIMS deposition occurred when the voltage was just high enough to enter the highly ionised deposition regime. In this work, we compare the time evolution of vanadium HIPIMS plasma with the previous results for titanium. Information about the time evolution of electron temperature and charge states is presented.

The introduction of spatially resolved sampling enhances understanding of processes underlying the time evolution observed. The results are used as a foundation for a model that could be used to find optimum regimes for deposition.

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POSTER PRESENTATION

HIPIMS vs DCMS technology to produce tungsten coatings for fusion applications

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Carbon-based materials have been used in thermonuclear fusion devices for a long period. The major drawback of a plasma-facing wall with carbon armour, such as graphite or Carbon Fiber Composite (CFC), is their chemical erosion and dust formation under hydrogen bombardment. Moreover the re-deposition of carbon layers containing tritium represents a serious safety concern. The use of W coatings deposited on carbon based materials can allow to overcome these problems. The employment of Tungsten as an armour material is convenient because of its high melting temperature, good thermal conductivity, low tritium retention, low sputtering rate. W films were deposited by various methods on carbon substrates: Vacuum Plasma Spray (VPS), Chemical Vapor Deposition (CVD) and Physical Vapor Deposition (PVD) techniques. However, because of the extreme working conditions (neutron bombardement, thermal cyclic fatigue, carbon diffusion, plasma erosion) at present none of the proposed deposition techniques is completely successful. Among the different technologies, HIPIMS seems to be the most promising because it permits to deposit high quality and ultra-dense coatings with highly controlled microstructure and residual stress. This work gives a comparison between the properties of several W coatings obtained by the conventional Direct Current Magnetron Sputtering (DCMS) and High Power Impulse Magnetron Sputtering (HIPIMS). The used deposition systems have been custom designed and built. In particular, the used HIPIMS generator is the Huettinger TruPlasma Highpulse one. In front of the 4 inches HIPIMS cathode a single rotating tree was mounted providing different target substrate distance (TSD) position between 50 and 200 mm. The substrate can be heated up to 800 °C and biased up to 1200 V. The W coatings have been grown on graphite, metal sheets and Si substrates changing progressively the sputtering parameters to optimize the process to obtain the desired thermodynamically stable body-centered-cubic form of metallic tungsten (alfa-W). The tungsten coating final phase depends on substrate bias and temperature, deposition pressure, residual gas species. Multilayered films were produced using Mo as a glue layer because tungsten and carbon can form metallic brittle carbide phases. Furthermore Mo compensates possible thermal expansion mismatch. The crystalline structure and orientation of the obtained film is analyzed by X-ray diffraction. The crystallographic parameters are evaluated using MAUD (Material Analysis Using Diffraction).
Their morphology and thickness were determined by electron scanning microscopy. Adhesion properties were evaluated by scratch tests and thermal shock tests. Hardness was estimated by Vickers indentation.

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POSTER PRESENTATION

HIPIMS deposition of TiAlN films on microforming die and its tribological properties in progressive micro-deep drawing

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Over the last two decades, microforming technology has been contributing on reducing the cost of mass production for micro to sub-millimeter range components. In view of the remarkable increase of surface area to volume ratio with miniaturization of the process dimension, the frictional behavior appears to become more dominant for the for ming process. Especially, from the standpoint of dirt handling, contamination of products, and unstable formability, dry forming process is strongly required. Thus, the activities to applying the hard film coatings on microforming die substrates are gradually increasing.

Technical issues on the hard film coatings for microforming are the remarkable improvement of the tool life to reduce the cost of maintenance. In general, to enhance the tool life, sufficient high wear toughness and low friction are required. However, additionally in micro scale forming die, the dimensional accuracy and the uniformity of the film thickness and mechanical properties also strongly influences on the durability of the forming tool. Moreover, to obtain the good tribological performance and stable formability, fine smooth surface would be required for the microforming die surface finishing.

As overviewed by many researchers, high power impulse magnetron sputtering (HIPIMS) deposition films has the advantageous features for the forming tool life, such as dense and smooth surface, and three-dimensional uniformity on complex-shaped substrates. Furthermore, since the number of the process parameter of HIPIMS enhances the controllability of the film property, the optimization, which corresponds to the different kind of die shape and forming process, could be possible. However, the availability and practical advantages of HIPIMS deposition films for the microforming die is not well discussed.

Within this background, this study aims to clarify the applicability and practical performance of HIPIMS coating films for microforming die. The fundamental studies on the effect of pulsing characteristics, such as pulsing frequency and duration, process gas pressure, and bias voltage, on the uniformity of the film properties are investigated. Based on this investigation, TiAlN film deposition using HIPIMS is applied to the practical micro-deep drawing die. Furthermore, in order to investigate the practical durability of this film, the progressive micro-deep drawing tests up to 1000 shots are carried out with the ultra-thin stainless steel foils of 50µm thickness. By the evaluation of the transition of forming force and the forming die surface, applicability of HIPIMS deposition films on microforming dies are discussed.

POSTER PRESENTATION

Titanium carbide oxide and nitride HIPIMS and DCMS processes compared from the OES point of view

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The aim of this study is to relate the effect of high power impulse (HiPI) and direct current (DC) power supplies in the deposition via magnetron sputtering (MS) of titanium composites such as TiC, TiO_2 and TiN on composition, structure and mechanical properties with optical emission spectroscopy in industrial conditions (target size 12" × 4.9").

Titanium carbide HIPIMS deposition process from a titanium target in acetylene (C_2H_2) reactive atmosphere shows the presence of Hi emission line in optical emission spectra that increases in intensity as a function of peak power and acetylene flux. No hydrogen lines can be observed in DC process. Moreover HIPIMS coatings show lower oxygen content. On the other side the hydrogen content in HIPIMS films for the same C/Ti ratio is always lower than for DC and hardness and conductivity are higher ($\rho < 400\ \mu\Omega$ cm). Titanium oxide HIPIMS deposition process in oxygen reactive atmosphere shows the presence of double charged oxygen ions emission lines that are increasing in the hysteresis going from metal to poisoned mode. On the other side the HIPIMS coatings show a refractive index always higher than DC and the possibility to obtain rutile and anatase phases in a controllable way. Also in the case of titanium nitride the HIPIMS deposition process presents very different emission line spectra and allows to obtain hard coatings with no substrate biasing.

These results will be presented and discussed and the relation with coatings properties with optical emission spectra will be further investigated through tailored experiments. In particular the presence of reactive gas ions as strictly related to the sputtered species will be pointed out comparing OES in HIPIMS and DC sputtering of a titanium target in oxygen and nitrogen reactive atmospheres to that of a carbon target sputtering. Hydrogen dissociation will be evaluated comparing OES of C_2H_2, CH_4 and H_2 reactive atmospheres with a carbon target. Furthermore, changes in electrical conductivity of a-C/TiC depending on the reactive gas C_2H_2 and CH_4 will be shown. Oxygen content in titanium carbide and nitride HIPIMS and DC coatings will be compared.

POSTER PRESENTATION

Chromium nitride (CrN) thin films deposited by MPPMS for protection of different steel substrates

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Chromium nitride (CrN) thin films are extensively used for steel protection due to their high wear resistance, low coefficient of friction and good corrosion resistance. During the last decades, transition metal nitrides have been commonly deposited by either cathodic arc evaporation (CAE) or dc – magnetron sputtering (dcMS) techniques providing good quality coatings for different applications. However, the emission of macroparticles (during CAE discharge) or the low ionization degree of the sputtered particles (during dcMS discharge) leads to a defective and porous film structure which can compromise the final coating properties. To overcome these problems, recently developed modulated pulsed power magnetron sputtering (MPPMS) technique has been used for coating depositions.

CrN films were reactively sputtered in Ar/N_2 atmosphere by MPPMS and deposited on high speed steel (M3:2) and AISi 316L stainless steel disks and silicon wafers. N_2/Ar ratio was varied from 0.1 – 1.5, in order to obtain different morphologies and crystal phases.

Microstructure and thickness analysis were performed by scanning electron microscopy (SEM). Different crystallographic structures were determined by X-ray diffraction (XRD) using a Cr Kα radiation. SRV Tribometer was used to simulate reciprocating sliding motion for the evaluation

of friction coefficient and wear resistance of different deposited coatings. Corrosion tests were carried out by potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) techniques in NaCl (0.06M) solution.

POSTER PRESENTATION

Investigations of very short pulse sequences in HIPIMS mode for reactive deposition of Silica

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Silica coatings are used besides optical applications as insulator for electric and sensor applications. For these applications it is most important to produce defect-free films with high field strength. Since reactive sputtering of silica needs a process control and leads to a low deposition rate, a PECVD-process was investigated. Preferably for PECVD processes the power supply is connected to the substrate, but in the case of depositing an insulator mid frequency or rf–sputtering is mostly used. In the presented case the plasma source is a HIPIMS driven target.

For high rate deposition of silica a PECVD-process a Cyprium power supply (oscillatory plasma) was used. The investigations were carried out using TMS as precursor, a carbon target as plasma source. The peak current density for the processes was in the range of 0.1 A/cm^2. The Current-voltage characteristics for different pulse sequences and charging voltages are discussed as well as the deposition rate in dependence of the working conditions (gas flow, working pressure, and average power). The insulating properties of the films were characterized by their critical leakage field strength.

POSTER PRESENTATION

Nb coatings for superconducting RF applications by HIPIMS

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High Energy particle accelerators such as the LHC at CERN are essentially based on two key elements: RF cavities to accelerate the particles, and magnets to steer the beams. In order to achieve state of the art performance, these devices are often made with superconducting materials. RF accelerating cavities are usually made of bulk niobium, which guarantees excellent performances with the drawback of high material cost and requiring very sophisticated manufacturing technologies. CERN has developed since the 1980s the technology of producing accelerating cavities made of a copper substrate coated with a thin niobium film, which is sufficient to carry the superconducting RF surface currents. The coating is usually performed by magnetron sputtering in UHV, and cavities produced this way are currently em-ployed in the LHC. Nb/Cu cavities suffer however from the drawback that their quality factor decreases (ie the power losses increase more than quadratically) with increasing the accelerating field, contrary to theoretical predictions and what is actually seen in bulk niobium cavities. The origin of this phenomenon is unclear at present, but one possible cause is related to the imperfect microstructure of magnetron sputtered films. Nb coatings by HIPIMS may thus improve the RF quality of accelerating cavities, using substantially similar coating hardware.

Test coatings have been performed at Sheffield Hallam Universtity, in order to study the correlation between coating parameters such as the current density and the pulse duration with ionization efficiency. Characterisation by SEM, TEM, and XRD showed the development of stronger texture, improved crystallinity and smoother coating morphology with higher ionisation. In parallel, RF cavities and test samples have been coated at CERN using appropri-

ately scaled coating parameters, and their superconducting properties, microstructure and texture have been characterized. The first results of this study are reported in this paper.

POSTER PRESENTATION

The structure and tribological properties of tungsten containing hydrogenated diamond-like carbon coatings

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Tungsten containing hydrogenated diamond-like carbon coatings were deposited with unbalanced magnetron sputtering technology. The effect of WC target power on the structure and tribological properties of W-C:H coatings were studied. Microstructure of the coatings were analyzed by Raman spectroscopy. Hardness and Young's modulus were tested on CSM Nano-hardness tester. Tribological properties of the coatings with different WC target power were studied on ball-on-disk tester. The result show that all of the coatings have typical diamond-like structure. The hardness of the coatings increase gradually as increase of WC target power at first, and then it decrease. Friction coefficient and wear lifetime also changed obviously with increase of WC target power. The mechanical and tribological properties of W-C:H coatings were strongly influenced by WC target power. It is important to control WC target power at a proper value to prepare coatings with excellent properties.

POSTER PRESENTATION

Titanium-doped MoS_2 lubricating coatings for space precision ball bearings

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708C Ball bearings were made of 9Cr18 steel which had precision of class 4, MoS_2-Ti composite coatings were deposited on inner and outer races of ball bearings by unbalanced magnetron sputtering system, and bearing cages were made of PTFE-based self-lubricating polymers. Bearing's tribological torques as a function of storage time in high humidity environment were studied by using LHU-2 thermal & humidity test chamber and Bearing 2000 torque measurement device. Developing bearing vacuum performance test rig to research bearing's torque as a function of running time, bearings were dismantled after test and using XPS to analyze the surfaces of bearing's races and balls. The results show that storied in the environment of 30 ˚C, 85 % RH, bearing's running torques increased proportionally as the storage time went on.

When run-in the bearings which were storied for 440 days, the start and average running torques of bearings could come back to the level which was the same as the test start, but the fluctuation of bearing's average running torque increased. Running in one direction at the speed of 650 r/min, in vacuum environment (~10 – 4 Pa) and ambient temperature, tested bearings showed good running performance with low friction torque varied only between 1 g • cm ~ 2 g • cm until to the test ended at 1.872 × 108 revolutions. XPS analysis showed that astable and sustainable solid lubrication system was established among bearing's races, balls andcages.

POSTER PRESENTATION

Overcoming HIPIMS deposition rate limitations by hybrid RF / HIPIMS co-sputtering and its relevance for NbSi films

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NbSi is an Anderson isolator widely used in low temperature applications such as bolometers and far-infrared detector cameras.[1] It is a very interesting system due to its adjustable critical temperature Tc that can be regulated between a few mK and a few Kelvin. The critical temperature of the Nb_xSi_1-x alloy can be easily controlled through the Nb composition *x* during the co-deposition process of Nb and Si.

The major requirements of NbSi deposition process are its reproducibility and uniformity on large area substrates, since the bolometers' complexity is continuously growing with increased number of the microstructured pixels needed for THz-range cameras. Furthermore, thicker and denser films might be of interest for next generation cameras.

Successful NbSi deposition can be achieved by magnetron co-sputtering leading to highly controlled plasma thin film processing. The resulting samples are amorphous alloys commonly obtained operating the magnetrons at very low pressures, as low as 0.06 Pa. Enhancing the deposition rate at such low pressure is challenging for both conventional and HIPIMS driven magnetrons. An alternative solution is the use of hybrid RF / HIPIMS co-sputtering, on which focuses this communication. The dual magnetron plasma reactor designed at CNRS-IEF presents excellent characteristics in conventional PVD operation. Implementing the HIPIMS power supply with pre-ionization system [2] instead of a DC power supply proved undoubtedly its efficiency in the same reactor. The pre-ionization appears critical at very low pressure when only one target is HIPIMS driven.

Moreover, extremely interesting results are obtained when the hybrid HIPIMS/RF co-sputtering is used showing a beneficial alternative to conventional deposition of NbSi films. The low temperature measurements demonstrated surprisingly sharp transitions with excellent responses. Moreover, from the set of experiments performed by keeping constant the HIPIMS average power, pulse width and repetition rate on one cathode, and by changing the RF power on the other magnetron cathode, it gave new insights into the elementary mechanisms sustaining the plasma at very low pressure. Comparisons with conventional magnetron RF /DC co-sputtering showed the interest of the hybrid RF / HIPIMS solution with equivalent deposition rates but improved film properties.

These successful results open new ways for further applications in cryo-material deposition.

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POSTER PRESENTATION

The structure and mechanical properties of Cr_2N coatings deposited by HIPIMS technology

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Cr_2N coatings were deposited by HIPIMS technology. The compositions, morphology, mechanical and tribological properties of Cr_2N coatings were studied. The compositions and morphology of the coatings were analysed by XRF and SEM. The hardness and Young's modulus of the caotings were measured with CSM nanohardness tester. The tribological properties of the coatings were evaluated by CSM ball-on disk tester. The result shows Cr_2N coatings deposited by HIPIMS are more denser, harder that that deposited by dcMS. The hardness of the coatings reach 24.6 GPa. The sliding wear tests of Cr_2N coatings sliding against different ballmaterials shows the lowest COF of 0.48 when sliding against Al_2O_3 ball and extremely low wear rate of 10^{-8} mm^3 $N^{-1}m^{-1}$ against 100Cr6 ball.

POSTER PRESENTATION

ZrSiN and NbN coatings deposited by HIPIMS for hard coating corrosion protection on aluminum

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The aim of this work is to analyze the main differences in the coating obtained by two different magnetron sputtering methods: the conventional Direct Current Magnetron Sputtering (DCMS), and the recently developed High Power Impulse Magnetron Sputtering (HIPIMS) used to deposit single NbN and ZrSiN layers and nano-structured NbN – ZrSiN multilayers. These innovative nitrides films deposited in a reactive sputtering atmosphere have been used to cover the aluminum 7075 alloy substrate for hard corrosion resistance application. Many samples have been deposited, varying nitrogen flux and substrate polarization. OES and current waveform were used to highlight the different reactive regimes and in the hysteresis.

Compositional analysis were carried out by RBS and cross-section SEM investigation was used to highlight coating morphology and the presence of defects. Moreover the differences between the HIPIMS and DC and between the substrate bias values were observed on crystal structure by XRD, on coating resistivity by four point probes measurement and on mechanical properties by nano-indentation and microscratch test. Electrochemical impedance spectroscopy and salt-spray test were used to evaluate the corrosion resistance of the different coatings.

POSTER PRESENTATION

Tungsten coatings by HIPIMS as plasma facing material for nuclear fusion reactor applications

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The constant increase in the world population and in the development degree leads to a high energy consumption. The energy solution is a long standing problem which requires innovative solutions. Nowadays, in combination with other sustainable sources of energy, fusion energy can become within the next two decades a real alternative to fossil fuels. However, the lack of materials able to withstand the severe radiation conditions (high thermal loads and atomistic damage) expected in such reactors is a bottle neck for fusion to become a reality.

The main requisite for plasma facing materials (PFM) is to have excellent structural stability since severe cracking or mass loss would hamper their protection role which turns out to be unacceptable. Additional practical requirements for PFM are among others: (i) high thermal shock resistance, (ii) high thermal conductivity (iii) high melting point (iv) low physical and chemical sputtering, and (v) low tritium retention.

W has been proposed to be one of the best candidates for PFM for both laser (IC) and magnetic (MC) confinement fusion approaches. However, works carried out up to now have identified some limitations for W which have to be defeated in order to fulfill specifications [1, 2, 3]. Nowadays, the capabilities of ultrafine grain and nanostructured materials for nuclear fusion reactor applications are being investigated [4].

We report on the optimization of the HIPIMS parameters for deposition of nanostructured tungsten coatings with a thickness in the micro-meter range. To this aim, we study the influence of HIPIMS parameters (target potential and pulse frequency) on the W coatings morphology, stress state and mechanical properties. For optimized parameters, Scanning electron microscope (SEM) images show that coatings consist of nano-columns with an average diameter of around 70 nm and a cylindrical shape that grows perpendicular to the surface substrate. X-Ray diffraction (XRD) studies show that coatings are pure α-phase and polycrystalline. Optimized coatings show good adhesion to the substrate. Nanoindentation tests evidence that the coatings hardness is as high as ~ 14 GPa.

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POSTER PRESENTATION

Optimization of HIPIMS photocatalytic titania coatings on polymeric substrates

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Titanium dioxide in its anatase form is widely used in photocatalytic applications due to its high photocatalytic activity, stability and low cost. Titania coatings directly deposited by conventional magnetron sputtering tend to have an amorphous microstructure. For the anatase structure to develop, substrate heating or post-deposition thermal treatment is usually required, with the anatase crystal phase generally forming at temperatures in excess of 400 °C. This precludes the choice of thermally sensitive substrate materials for the photoactive coating.

Depending on the nature of the driving voltage waveform, high power impulse magnetron sputtering (HIPIMS) has been shown to deliver a relatively low thermal flux to the substrate, whilst still allowing the direct deposition of crystalline titania coatings. Consequently, this technique offers the potential to deposit photocatalytically active titania coatings directly onto polymeric substrates and, therefore, open up a range of new applications. In the present work a range of titanium dioxide thin films were deposited by HIPIMS onto glass substrates in order to study the influence of various process parameters, such as pressure, pulse frequency and pulse duration on coating structure and photocatalytic properties. The photocatalytic properties of the coatings were assessed by their ability to degrade the organic dye methylene blue under UV and fluorescent light irradiation. The degradation rate of methylene blue was calculated by measuring its absorption peak height at 665 nm in continuous mode under UV / fluorescent light source. The hydrophilic properties of the coatings were also investigated by measuring the contact angle of water droplets on the coating surfaces.

Optimised coatings then were deposited onto a range of polymeric substrates, such as polyethylene terephthalate (PET), polycarbonate, polypropylene, etc. to assess the suitability of using this method for high-energy, low-temperature deposition of photoactive titania coatings.

POSTER PRESENTATION

Growth of carbon – tungsten nanocomposites by high power impulse magnetron sputtering from compound targets

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High power impulse magnetron sputtering (HIPIMS) enables the implementation of the advantages of the Ionized Physical Vapour Deposition paradigm (high proportion of ionized species and control over particle energy) for conventional MS setups. Therefore it triggers not only scientific but also industrial interest.

Here we present a basic study of the processing-structure relations of C-W nanocomposite films grown in pulsed direct current (DC) MS and HIPIMS modes. A 3" C-W (~ 10 at. %) compound target has been chosen over single component targets for the investigation of principal target surface interactions initiated by HIPIMS. Carbon reference films have been grown from pure carbon targets for comparison. The deposition time for all films was identical for easy comparison of growth rates. The film areal density has been determined by Rutherford backscattering spectrometry and nuclear reaction analysis. The film structure has been studied by Raman spectroscopy, X-ray diffraction and transmission electron microscopy.

For pure carbon targets the duty cycle for a fixed power was limited by extensive arcing. In contrast, a stable HIPIMS regime can be established for the compound target with a duty cycle as low as ~ 1 %. Resulting films consist of WC nanoparticles embedded in a carbon matrix. The film areal density of tungsten in the C-W films shows only a small drop with decreasing duty cycle (transition from pulsed DC to HIPIMS mode). Despite different sputtering modes (by Ar ions in pulsed DC, by a mixture of Ar/W ions in HIPIMS), the film areal density of carbon does not exhibit any significant changes and the typical drop in growth rate going from pulsed DC to a HIPIMS discharge has not been observed. Collisional computer simulations using TRIDYN, which take into account changes in surface chemistry and composition during sputtering, demonstrate a considerable sputter yield amplification of carbon when irradiated with a mixture of Ar/W ions. This is in-line with the observed stable carbon film areal density, which can be attributed to the compensation of the change in sputtering ion composition by sputter yield amplification due to W-enrichment of the target surface.

POSTER PRESENTATION

Ellipsometric characterization of transparent nickel oxide deposited by reactive DC magnetron sputtering and HIPIMS

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The organic photovoltaic cells (OPV) up to now convert 5 % of the solar spectrum and last reports predict that efficiencies as high as 10 % can be achieved. For the classical OPV, charge carrier transport from the photosensitive polymer is performed by incorporation of organic semiconductors which are in contact with metallic electrodes. The interfaces between metal and organic components as well as the nature of organic semiconductors play an important role for the effective charge transport and degradation processes. Limitating the sensitivity of polymer materials becomes the path for development of hybrid OPV. One possible way is to use inorganic semiconductors. Metal oxide semiconductors as protective layers between metal electrodes and polymers, are interesting candidates for achieving charge transport and avoiding the organic-metal interfaces. We propose the NiO as p-type semi-conductor to ensure charge carriers and electron/hole blocking layers. The characteristic wide gap energy permits one to achieve high transparency for thin films of NiO.

We have deposited transparent p-type semiconductive NiO thin films on the conductive glass by reactive DC magnetron sputtering and HIPIMS.

In a previous work[1] we have shown that depending on the oxygen content, NiO grows along preferential orientations: either the most dense [111] direction for low oxygen percent or [200] for higher oxygen content. We have also highlighted the same behaviour by HIPIMS which can be managed by the frequency and/or the pulse duration[2,3].
In this study, we have investigated the dependence of optical properties of NiO films using spectroscopic ellipsometry (1.5 – 5.0 eV range). NiO films were obtained when the oxygen content was 15 % in DC case and 9 % in HIPIMS case. Refractive index n, extinction coefficient k, and gap energy of the NiO films were determined with a refractive index gradient changed along the film growth direction whereas the NiO dispersion functions were described using the Tauc Laurentz model…

The general trends identified for a NiO film with a typical thickness range from 150 to 250 nm shows a decrease of the refractive index and an increase of the transparency range when going from the bottom to the top of the layer 4.

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POSTER PRESENTATION

The reactive high power impulse magnetron sputtering process for the synthesis of CF_x thin films using CF_4 and C_4F_8

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Reactive high power impulse magnetron sputtering (HIPIMS) processes have been explored for the growth of fluorine-containing amorphous carbon thin films (CF_x, $0.15 \le x \le 0.35$) comparing the precursor gases tetrafluoro-methane (CF_4) and octafluorocyclobutane (c-C_4F_8). The fluorine content of the thin films was controlled by varying the partial pressure of the F-containing gases between 0 mPa and 110 mPa, while keeping the deposition pressure of 400 mPa and the substrate temperature of 110 °C constant.

The HIPIMS processes were investigated with time averaged positive ion mass spectrometry and the resulting thin films were characterized regarding their composition, chemical bonding and microstructure as well as mechanical properties. The experimental results are compared to results obtained by first-principles calculations based on DFT. The calculations on the most abundant precursor fragments provide data on their relative stability, abundance, and reactivity. Positive ion mass spectrometry on the C_4F_8 plasma showed, next to inert gas ions, an abundance of CF^+, C^+, CF_2^+ and CF_3^+ (in this order), whereas only CF_3^+ exceeded the Ar^+ signal in case CF_4 was used. Judged by TEM, all synthesized CF_x films are amorphous. Results from X-ray photoelectron spectroscopy indicate a graphitic nature for films with fluorine contents below 24 at % and a polymeric structure for films with fluorine contents above 26 at %.

This is mirrored in their mechanical properties as the hardnesses decreases steeply for these CF_x thin films exhibiting a polymeric structure.

POSTER PRESENTATION

Compressive stress generation through adatom insertion into grain boundaries in low mobility metal films deposited by high power impulse magnetron sputtering

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High power impulse magnetron sputtering has proven to be a versatile ionized PVD technique where coating microstructures and properties can be tuned by changing the bombarding condition through changing the pulse power. In this work we use this versatility to investigate the causes of intrinsic stresses in polycrystalline films. Generation of intrinsic stresses in refractory metals or other high melting temperature materials deposited at low temperature have previously been considered originate from two additive stress sources; tensile stresses generated from attraction between grains over underdense grain boundaries and compressive stresses generated by point defects introduced by energetic bombardment. Compressive intrinsic stresses has also been suggested to be generated by adatom diffusion into grain boundaries for deposition conditions with highly mobile adatoms, i.e. homologous temperatures > ~ 0.2. HIPIMS discharges where used to tune the energetic bombardment conditions during deposition Mo films at low homologous temperatures and achieve atom mobility in the near surface layers while still freezing the state of the bulk of the film. This allowed us to investigate the stress-microstructure relationship post deposition.

We found that the film stresses ranged from -3 to +0.2 GPa while maintaining a nearly constant stress free lattice parameter. This implies that the compressive film stress is not caused by defect creation in the grains but primarily by grain boundary densification, a scenario also supported by a correlation between large compressive film stresses and high film densities. Based on this we suggest a growth mechanism active during the energetic bombardment conditions present in HIPIMS film deposition leading to smooth dense films exhibiting compressive stresses.

POSTER PRESENTATION

High power impulse magnetron sputter deposition of ITO and AZO thin films

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High power impulse magnetron sputtering (HIPIMS) is a promising physical vapour deposition (PVD) technique with a number of unique properties, such as an ultra-dense plasma and a high degree of ionization of the sputtered atoms. In this work, HIPIMS was used to deposit In2O3:Sn (ITO) and ZnO:Al (AZO) thin films on polyester substrate rolls. The process was controlled by varying the electrical parameters and the oxygen flow based on the optical emission line intensities measured by Plasma Optical Emission Spectroscopy. A photodiode with an oscillograph was used to study the kinetics within a single power pulse. It was found that HIPIMS reduces the mechanical tension of the films and increases the flexibility, compared to the films deposited by regular DC sputtering. There was no oxide growth in the erosion zone of the target, which is an advantage for long deposition processes. The challenge still to be solved is the relatively low dynamic deposition speed, arising from the inability of the power source to provide stable arc-free sputtering conditions at sufficiently high power levels.

POSTER PRESENTATION

Uniform, adhesive, robust Cu/TiO_2 DCP and HIPIMS sputtered films inducing fast bacterial/viral inactivation under low intensity solar irradiation

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Sputtering of Cu[1-2] and TiO_2/Cu[3-4] films on textile by conventional pulsed direct current magnetron sputtering (DCP) and highly ionized pulsed plasma magnetron sputtering (HIPIMS) was performed to inactivate E. coli (Gram-negative) and methicillin resistant staphylococcus aureus (MRSA, Gram-positive). Low intensity sunlight with an intensity of 0.5 – 1 % of solar irradiation AM1 were able to accelerate the bacterial inactivation. This is important when precluding the formation of biofilms pumping infectious bacteria in hospitals, schools and other public places since biofilms remain active for long-times and are a source of pathogens. Film surface characterization was carried out by X-ray fluorescence (XRF), profilometry, electron microscopy (HRTEM), AFM, contact angle CA and X-ray photoelectron spectroscopy (XPS). By highly ionized pulsed plasma magnetron sputtering (HIPIMS) inducing an increased M^+-ions energy and high charge density of ~10^{18-19} e-/m^3, a coating ~38 nm thick inactivated *E. coli* within 10 minutes by way of a TiO_2 60/Cu 40 atomic percent target. By DCP sputtering the bacterial inactivation time was similar to the one induced by HIPIMS times but a coating ~ 600 nm thick was required. Under actinic light radiation, samples deposited by pulsed magnetron sputtering time of 150 s induced short inactivation times of ~10 min. This sample presents the most suitable structure-reactivity for the Cu-clusters on the TiO_2. The bactericide action under light was due to a synergic effect in the TiO_2/Cu layers since longer inactivation times were observed when sputtering separately TiO_2 or Cu.

DCP sputtering leads to M^+ ionization >> 5% and a charge density ~1016 e-/m^3 HIPIMS leads to Cu ionization of ca. 70 % with a charge density ~10^{18-19} e-/m^3 and with a power per pulse of 1750 W/100 microseconds compared to the DCP power per pulse of 62.3 W/10 microseconds.

The effective antibacterial action by the HIPIMS film was possible due to the higher applied V since a higher V increases the transition M → M+.

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POSTER PRESENTATION

Optical emission spectroscopy of aluminum nitride thin films deposited by pulsed laser deposition

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In this work we study the Aluminium Nitride plasma produced by Nd:YAG pulsed laser, (λ = 1064 nm, 500 mJ, τ = 9 ns) with repletion rate of 10 Hz. The laser interaction on Al target (99.99 %) under nitrogen gas atmosphere generate a plasma which is produced at room temperature; with variation in the pressure work from 0.53 Pa to 0.66 Pa matching with a applied laser fluence of 7 J/cm^2 The films thickness measured by profilometer was 150 nm. The plasma generated was at different pressures was characterized by Optical Emission Spectroscopy (EOS) From emission spectra obtained ionic and atomic species were observed. The plume electronic temperature has been determined by assuming a local thermodynamic equilibrium of the emitting species.

Finally the electronic temperature was calculated with Boltzmann plot from relative intensities of spectral lines. Moreover Surface Acoustic Wave (SAW) devices with a Mo/AlN/Si_3N_4 configuration have been fabricated, employing AlN-buffer and Mo Channel.

POSTER PRESENTATION

Carbon ion production using a high-power impulse magnetron sputtering (HIPIMS) glow plasma

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Carbon ions are significantly produced in a HIPIMS glow plasma by using a self-made magnetron sputtering plasma source with a target diameter of 150 mm. The magnetic flux density on the target and its distribution are changed by arranging the configuration of the permanent magnet, which is formed as a concentric circle, and the set of the magnet is unbalanced. The important issues to significantly produce carbon ions are (1) a high target voltage so as to obtain a significantly high argon energy, (2) a high density argon plasma and (3) no arc transition. In order to enhance the ionization of the sputtered carbon-target species, first of all, magnetic flux configuration is varied so as to find a stable glow plasma in HIPIMS, and second, a pulsed source voltage with a short duration is applied to the target in order to apply high voltage to the target without transiting to an arc discharge. Thus, a source voltage over 2000 V can be applied to the target and a glow voltage over 1200 V at the end of the pulse of 5.5 µs. The plasma position moves outward on the target by increasing the number of the inner magnet. As a result, the plasma edge approaches to the grounded plate set around the of target. Hence, an arc easily occurs at a lower voltage with an increased number of the inner magnet. In order to confirm the carbon ion production, the optical emissions near at the target are observed using a spectrometer; typically, wavelengths of carbon ions and argon ions of 388 nm and 435 nm, are observed, respectively. The increase in the intensity of the optical emission is brought by the increases of the consumed power density in the target. The emission intensities of argon and carbon ions have a proportional relationship to the consumed power.

As a result, the emission intensity of the carbon ions is proportional to that of the argon ions. This suggests that the production of the carbon ions is based on the electron impact ionization occurs to produce the carbon ions in the HIPIMS glow plasma.

POSTER PRESENTATION

The blood platelet behavior of titanium-copper films by high power pulsed magnetron sputtering

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Surface modification with high-power glow discharges is a promising physical vapor deposition (PVD) technology for industrial usage. A metal ion density higher than 1018 m^{-3} can be obtained due to a high power input in the plasma.

This article reports the titanium-copper films generated in a high power pulsed sputtering (HPPS) plasma with a magnetron sputtering system, where a substrate is set nearby the target for positions of 90 mm. The copper ion release behavior were investigated via a typical immersion test. The electrical- and optica characteristics are investigated. The phase composition, structure, and concentration of elements were investigated via X-ray diffraction and X-photoelectron energy spectrum. The blood compatibility is investigated using in vitro releasing of nitric oxide experiment and blood platelet adhesion.

The results are compared with characteristics by direct current magnetron sputtering (DCMS) process.

POSTER PRESENTATION

Deposition of nickel by inductively coupled impulse sputtering (ICIS)

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Inductively coupled impulse sputtering (ICIS) is the latest development in the field of highly ionised plasma processes. ICIS combines the advantages of RF-ICP, in that it can sputter materials that are typically challenging in conventional magnetron based systems such as magnetic materials, with the advantages of highly ionised metal plasma.

By generating the plasma inside a high power pulsed-RF coil in combination with a magnet free high voltage pulsed cathode it is possible to eliminate the need for a magnetron. This feature enables the generation of high density highly ionised metal plasma of magnetic material.

This paper gives an overview of the plasma properties and magnetic properties of the coating. The setup comprises of a 13.56 MHz pulsed RF coil operating at a frequency of 500 Hz and a pulse width of 150 µs, which results in a duty cycle of 7.5 % . A pulsed DC voltage of 1900 V was applied to the cathode to attract Argon ions and initiate sputtering. Optical emission spectra (OES) for argon and nickel species sputtered at a constant pressure of 14 Pa, show a linear intensity increase for peak RF powers of 1000 W – 4800 W. The influence of pressure on the process was studied at a constant peak RF power of 3000 W for pressures of 6 – 26 Pa. Argon neutrals rise linearly with increasing pressure.

Energy resolved Mass Spectroscopy results for Ni^{1+} show a non-maxwellian ion energy distribution with a peak at 20 eV. This represents a good value for ion surface mobility without inducing lattice defects. This can be useful for deposition onto high aspect ratio structures.

The Nickel deposition rate is 50 nmh^{-1} for a RF-power of 3000 W and a pressure of 14 Pa. The microstructure of the coatings shows globular growth. For lower process pressure the microstructure changes to dense columnar structures with dendritic features. Bottom coverage of unbiased vias with width 0.300 µm and aspect ratio of 3.3:1 was 15 % and for an aspect ratio of 1.5:1 was 47.5 %. Perpendicular growth can also be seen on side walls with densification on the bottom. Parameters for this coating are mean values from a power and pressure matrix. EDX measurements have shown that it is possible to deposit Nickel coatings that are not contaminated by induction coil material.

The magnetic properties have been examined by magneto optical kerr-effect spectroscopy MOKE. This technique utilises the kerr-rotation of a non-polarised laser to measure the hysteresis loop. For a 200 nm thick ICIS deposited coating the results are very close to that of bulk material.

The current work has given an overview of the plasma properties and deposition of nickel in an ICIS plasma. These results are very promising for the deposition of materials that have been difficult to utilise in conventional magnetron sputtering, specifically for magnetic materials.

The authors would like to thank Prof. Olinda Conde and Nikolay Polushkin from the University of Lisbon for their cooperation in the MOKE measurements.

POSTER PRESENTATION

Film deposition using a 1 inch-sized HIPIMS system

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Hard film coating with excellent tribological characteristics has been tried out and realized using HIPIMS technology. We have been engaging in a development of a coating process using a small-scaled magnetron sputtering source with a target size of 1 inch in diameter. This would be incorporated in a newly-developed semi-conductor production system, which is called »minimal-fabrication production system« [1]. The base of this technology is a film deposition on a half-inch sized substrate with a small-scaled plasma production method. In order to realize the plasma system, the issues below are considered;

1. The plasma generation system with a water cooling system
2. Suppression of the plasma radial expansion on the sputtering target, and
3. Facilitation of the expansion of the sputtered metallic species (atoms and ions) to the substrate.

A high magnetic flux density over 1000 G on the target at a source voltage less than 600 V at a gas pressure as low as 0.6 Pa, where no arc transition is observed. Copper films are prepared under these conditions. In order to enhance the deposition rate of the prepared films, a pulse duration as long as 600 µs at a repetition frequency of 100 Hz is employed. As a result, the deposition rate as high as 2.5 µm/min on a 0.5 inch silicon wafer is realized.

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POSTER PRESENTATION

Time- and space-resolved laser-induced fluorescence spectroscopy in a short-pulse HIPIMS discharge

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The velocity distribution function of the sputtered particles in the direction parallel to the planar magnetron cathode is studied by spatially- and time- resolved laser-induced fluorescence spectroscopy in a short-duration (20 µs) high-power impulse magnetron sputtering discharge.

The experimental evidence for the neutral and ionized sputtered particles to have a constant (saturated) velocity at the end of the plasma on-time is demonstrated. For Ti atoms and ions, the velocity component parallel to the target surface reaches the values of about 5 km/s, which is higher that the values typically measured in the direct current sputtering discharges before. This points out on the presence of a strong gas rarefaction significantly reducing the sputtered particles energy dissipation during a certain time at the end of the plasma pulse, referred to as »rarefaction window« in this work. The obtained results are in line with the data collected during the analysis of the plasma off-time previously carried out in Britun et al. Appl. Phys. Lett., 99 (2011) 131504.

The results are compared to the velocity distribution function width corresponding to the Ar metastable atoms present in the HIPIMS discharge (measured in the same direction), as well as to the recent time-resolved mass spectrometry results (measured perpendicularly to the target surface).

POSTER PRESENTATION

Angle- and time-resolved ion velocity distributions in HIPIMS

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The High Power Impulse Magnetron Sputtering System (HIPIMS) equipped with 2" in diameter target has been investigated by means of time-resolved mass- and energy-resolved analyser (plasma monitor) from Hiden Ltd., gridded retarding field energy analyser (RFEA) from Impedans Ltd. and so called modified Katsumata probe. All the methods allow to determine ion velocity distribution functions (IVDF) in forward direction to substrate as a function of retarding electric field. However, except plasma monitor latter methods are not able to resolve the mass of particles. The newly developed modified Katsumata probe uses a static magnetic field created by Sm-Co permanent magnets to intercept the most of plasma electron and pull them away back to the plasma bulk. Furthermore, the plasma monitor and the modified Katsumata probe are characterized in very small angular acceptance in comparison with the gridded RFEA. The high power impulse magnetron sputtering system was equipped with pure metallic targets (titanium or iron). As working gas a mixture of Ar and O_2 was used. The working gas pressure was ranging between 0.5 Pa to 5 Pa. All the diagnostic instruments were placed at position of substrate. All the measurements were carried out under the same conditions as the thin oxide films TiO_2 and Fe_2O_3 were deposited. A comparative study of all the aforementioned methods has been carried out.

Results clearly demonstrate an influence of angular resolution on measured IVDF. Unlike gridded RFEA the modified Katsumata probe was able to distinguish different groups of ions coming on the substrate in different times of plasma pulse. The gridded RFEA has angular acceptance more than 70 °C and ions reaching its input orifice originate from different direction unlike the modified Katsumata probe which accepts ions only from cone of a small solid angle. The temporally resolved investigation with plasma monitor revealed that sputtered particles reach a substrate later on. All the plasma diagnostic methods revealed significantly enhanced energy tail in ion velocity distributions measured in HIPIMS in contrast to dc magnetron or mid-frequency pulsed-dc magnetron. An influence of working gas pressure on velocity distributions of argon, metallic and reactive gas ions specifically on presence of high-energy tail is discussed. The plasma monitor proved that under certain plasma conditions appearance of double ionized sputtered and working gas particles can be observed.

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POSTER PRESENTATION

Deposition rate enhancement in HIPIMS without compromising the ionized fraction of the deposition flux

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We systematically investigate and quantify different physical phenomena influencing the deposition rate, a_D, of Nb coatings prepared by High Power Impulse Magnetron Sputtering (HIPIMS), and propose a straightforward approach for deposition rate enhancement through the control of the magnetron's magnetic field. The magnetic field strength at the target surface, B, of a 50 mm diameter magnetron was controlled by the application of paramagnetic spacers with different thicknesses in between the magnetron surface and the target. We found that lowering B achieved by the application of a 2.8 mm thick spacer led to an increase of a_D by a factor of ~4.5 (from 10.6 to 45.2 nm min^{-1}) when the discharge was operated at a fixed average pulse target power density (2.5 kW cm^{-2}). However, the ionized fraction of the deposition flux onto the substrate was found to be comparable, despite of a large difference in B-dependent discharge characteristics (magnetron voltage and discharge current). We show that the decrease in a_D commonly observed in HIPIMS (ranging from 33 % to 84 % in comparison with DC magnetron sputtering in the presented experiments) is governed by different physical processes, depending on the value of B: For high B, the back-attraction of the target ions towards the target is the dominant effect, while for low B the ion back-attraction, the sub-linear dependence of the sputtering yield on the ion energy, and the variation in material transport effects are all important. Finally, we offer a theoretical explanation for the observed results, demonstrating that the here-presented conclusions are applicable to all HIPIMS discharges in general.

POSTER PRESENTATION

Amorphous carbon matrix – carbon nanotube nanocomposites

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Developing new sensors on the basis of nanocomposite materials is highly promising. Materials comprising different nanostructures may be especially interesting due to the diversity of their functional possibilities. Carbon nanotubes (CNT) incorporated in a diamond-like carbon (DLC) matrix are a promising material because of its high electrical and thermal conductivity which will enhance the sensitivity of corresponding semiconductor devices. Nanotechnology research and development can enhance security capabilities by introducing new concepts and improvement the functionality of existing ones. However there is limited knowledge related to mechanisms of the corresponding nanostructure formation. The ability of varying growth conditions will contribute to advancing the functional diversity of materials for specific applications. Diamond like carbon (DLC) coatings consisting of carbon atoms are biocompatible due to their high corrosion- resistant properties, and therefore are an appropriate material for matrix (platform).

Nanotube-DLC matrix composite materials have been prepared from the gas phase by a direct current, glow-discharge plasma, whereby nanotubes formed spontaneously within the DLC matrix. At various stages of the growth the control of technological processes and plasma composition was conducted in real time. In contrast to the case of composites with specially-embedded defined nanotubes, in our case it is necessary to determine the qualitative composition of the obtained nanotubes. Thus the treatment of methods used for the growth of the nanocomposite is very timely, moreover such methods are not sufficiently developed.

We propose an approach whereby CNT and other nanostructures may be formed using a relatively simple and cost-effective technique of plasma-chemical deposition. The approach has a great potential as it enhances the conventional magnetron sputtering technology with an additional ion source, which offers a high flexibility in materials microstructure design. The equipment has a fully computerized control for supplying additional agents within the plasma thus allowing high experimental precision, which is crucial. Especially interesting is the possibility to improve the growth of nanotubes by using additional short high power magnetron impulses to inject within the plasma metal species such as Ni and Fe to promote CNT formation.

The multitude of nanostructures, their possible arrangement, and peculiarities of processes taking place on the nanostructure-matrix interface all give an opportunity for systematic experimentation with the objective of further development of advanced technology.

ORAL PRESENTATION

Ionization zones, plasma flares, self-organization, and the asymmetric ejection of particles in high power impulse magnetron sputtering

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Over the last couple of years, several groups reported on self-organized, traveling patterns observed in the plasma of magnetrons operating in high power impulse magnetron sputtering (HIPIMS) mode. Sideon fast framing and streak camera images indicate that each of the ionization zones, a.k.a. plasma spokes, emit a plasma flare away from the target. A model emerges where the large current seen in HIPIMS can be explained by locally breaking electron confinement at the edge of an ionization zone. The self-organization of ionization zones is affected by both the presence of atoms available for ionization, and electrons sufficiently energetic to cause ionization. Sputtered atoms as well as gas atoms contributed to the inventory of atoms available for ionization, and their presence is affected by the location and intensity of ionization zones. Such positive feedback mechanism and energy dissipation are typical ingredients for self-organized systems, which show features of chaos conditionally evolving into spatially symmetric and temporally periodic structures. Associated with those structures are current and particle transport, not only to the substrate but also near the plane of the sputtering target. We report on measurements of azimuthally asymmetric particle fluxes exhibiting patterns associated with traveling ionization zones. Side-on image of a Nb HIPIMS discharge in 0.3 Pa Ar, taken with an exposure time of 10 ns, 5 cm diameter target, 500 A peak current, 50 Hz. False color indicates intensity of emitted light, integrated over the visible spectrum.

For more information see
P. A. Ni, et al. Appl. Phys. Lett., 101 (2012) 224102.

ORAL PRESENTATION

Mechanism of the instabilities in HIPIMS discharge

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Recently an inhomogenity of the HIPIMS discharge has been reported[1,2]. In our previous papers we demonstrated and explained the influence of the power and pressure on the instabilities. Furthermore we investigated transition form stochastic to periodic behaviour[3]. In this contribution an explanation of the shape and mechanism of the instabilities is presented, based on the experimental observations. The experimental results of a 4 camera setup, photomultiplier tube data correlated with the biased flat probe are presented. A heuristic model combines a particle approach together with a global approach demonstrating that both violation of the β limit and a modified Rayleigh-Taylor instability could be responsible for the mechanism of the instability. Optical emission spectroscopy provides an understanding on the different emission profiles for different cathode materials.

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ORAL PRESENTATION

Global modeling of the azimuthally rotating structure in HIPIMS

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High Power Impulse Magnetron Sputtering (HIPIMS) is a novelIonized Physical Vapor Deposition technique, able to achieve an ultra dense plasma with a high ionization degree among the sputtered atoms. This is accomplished by applying a large bias voltage to the target in short pulses with low duty cycle.

HIPIMS discharges are characterized by high density plasma (peak electron density $10^{18} – 10^{20}$ m^{-3}) in a strong magnetic eld (100 mT), with highly energetic secondary electrons (500 – 1000 eV). The combination of these factors results in a discharge showing a vast range of instabilities ranging from MHz (i.e. modied two stream instability[1]) to 10 – 100 kHz range. Here we attempt the description of ionization zones breaking the azimuthal symmetry of the set up and rotating with about 10 % the E × B drift velocity in the same direction. This phenomenon has been observed by several authors[2, 3, 4] in dierent experimental set up. It is argued that these spoke-like structures determine the overall plasma density, carry most of the discharge current and are responsible for anomalous cross eld electron transport. It is therefore fundamental to understand their formation and relevance in order to characterize the system behavior.

During the on time of the bias, the current increases until it reaches a maximum then remains constant. Experimental observations[5] show the formation of a number of features rotating, that decrease in number until only one spoke remains. It is moreover observed that this spoke rotates with a constant angular velocity. We theorize that the conguration with one or more spokes depending on the power coupled to the discharge is indeed a »periodic equilibrium« conguration, to which the system relaxes to when it switches from Conventional DC Magnetron Sputtering to HIPIMS mode. We assume there is a single structure that rotates with a constant speed, during the plateau period of the current. These condition are experimentally observed by Hecimovic et al.[6] for a 0.17 Pa pressure Ar-Al discharge with -800 V bias and 100 A peak discharge current: the single structure shows stationary behavior after 170 s and rotates with Ω ≈ 80 kHz.

Since the discharge is sustained by very energetic secondaries and the electrons are far from equilibrium, we develop a global model that evolves the electron energy distribution function self-consistently with the rate equations for background gas and metal species. The volume average is performed *only* in the structure region and a net neutral flux term is imposed to model the spoke rotation at speed Ω *r*. Within the spoke region the species densities $n_e(\Theta)$ and $n_n(\Theta)$ are given a shape taken from a simple equilibrium 1D fluid model: this allows us to specify the net flux of neutrals even if the global model does not have an explicit spatial dependence.

The system consisting of the Boltzmann equation for the electron energy distribution function and rate equations for Ar and Al species is evolved in time through a pseudo-transit using a sti equation solver. The tracked densities $n_e(t)$; $n_{Ar}(t)$ and $n_{Al}(t)$ reach a steady state at physically meaningful values after only a few periods $T = 1/\Omega$.

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ORAL PRESENTATION

Dynamics of the fast – HIPIMS discharge during FINEMET-type films deposition

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The kinetic of the transient plasma particles remains an interesting field of study for understanding of the complex physico-chemical mechanisms involved in the sputtering and thin films deposition processes by sputtering method. The space and time evolution of the sputtered particles during the pulse plasma and in the afterglow period is of crucial interest for understanding of the HIPIMS mechanism of sputtering and deposition of various materials. The onset of the self-sputtering regime and the particles transport were investigated using electric measurements (cold and emissive probe), fast imaging, time-resolved optical emission spectroscopy and space- and time-resolved laser spectroscopy. $Fe_{73.5}Cu_1Nb_3Si_{15.5}B_7$ amorphous thin films have been deposited using the HIPIMS technique. The pulse voltage was set to a constant value of -1 kV, for relatively short pulses (4-20 µs) with a repetition frequency in the range of 0.1 to 2 kHz, attaining a maximum cathode power density of about 5 kW/cm^2. The influence of certain deposition conditions as working gas pressure, pulse duration, average power and target-to-substrate distance on the particle transport, which is related to deposition rate, plasma composition and thin films properties, have been studied.

The used plasma diagnostic methods provide valuable and complementary information about the particles kinetics and plasma-target and plasma-substrate interaction, respectively. They were successfully correlated for a better understanding of the processes that occur during films deposition and also for establishing an optimal set of discharge parameters for obtaining amorphous FINEMET-type thin films with excellent soft magnetic properties.

ORAL PRESENTATION

Spokes modelling by pseudo 3D PIC MCC

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Recently it has been found that spokes are rotating around the magnetron axis following the race-track of the target, especially in HIPIMS operation mode [1]. Many theories have been advanced to explain the origin of these structures based on ion impact onto the target, ionization waves, plasma instabilities, and the release of electron burst from the cathode. However, the comprehension of this phenomenon is very sparsely and self-consistent modelling can bring new insights, which is the purpose of this communication. In the last years we developed a Particle-in-Cell code dealing with plasma kinetics treated by Monte Carlo. Due to the fast variation of the voltage during the pulse, the reduced time-step required by the electron gyro-frequency and the stability criteria, the model was reduced to 2D. However, the spokes are intrinsic 3D phenomena and hence the third dimension has to be taken into account.

Numerical simulations show that the velocity drift $E \times B/B^2$ combined with the space charge repulsion lead to a non uniformity of the magnetron plasma along the azimuthal direction resulting in the creation of spokes [2]. To overcome full 3D models, a novel algorithm has been developed and successfully tested.

Indeed, all the test particles trajectories in PIC-MCC simulation are calculated in the real 3D space. Hence, the limiting point is the knowledge of the electric field in the azimuthal (y) direction. Using the electric field map obtained at a steady state by 2D modelling of the magnetron discharge in (x, z) plane, it is possible to self-consistently follow the particles in (x, y) and (y, z) planes, assuming that E(x, z) is constant (plateau of the pulse). This highly reduces the computation time and produces good qualitative results.

The obtained results, such as ion and electron 3D density maps and their projection along the azimuthal direction will be presented and discussed, as well as the effect of the ion mass.

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ORAL PRESENTATION

Kinetic modelling of an Ar-Cu preionized HIPIMS discharge

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High Power Impulse Magnetron Sputtering discharges are currently studied for advanced processing particularly for the deposition of thin film materials. In parallel to experimental studies and development of novel technologies and industrial applications a great deal of attention is focused on modelling and simulation of this type of discharge.

Monte-Carlo and Particle-In-Cell Monte-Carlo-Collision (PIC-MCC) numerical techniques are actively used to simulate the particle transport in the discharge, particularly, for studying its dynamics. It could be interesting for the understanding of the charged species organization, such as spoke instabilities which are actively studied in the last years. An important aspect of HIPIMS discharges is that they are characterised by non-equilibrium processes in particular for what concerns the electron energy distribution which presents a highly non-maxwellian tail despite the relatively high α ionization ratio which can be as high as $10^{-2} – 10^{-1}$.

The effect of this non-maxwellian and non-equilibrium character of the eedf (electron energy distribution function) in the magnetized part of the discharge and its consequences on the kinetic behaviour of the discharge can be captured by a non-equilibrium model associating the numerical resolution of the Boltzmann Equation (BE) for the magnetized part of the discharge (that we usually call magnetized negative glow) with a Collisional-Radiative Model (CRM) for neutral and ionic species created in the plasma. This type of model was already successfully used for the modelling of DC non reactive [1,2] and reactive discharges [3] and was demonstrated to be effective through the comparison of the CRM model results with experimental ones. This approach coupling BE and CRM within one model named OBELIX (Orsay Boltzman for Electrons and Excited Species) is also pertinent in order to emphasize the role of specific processes resulting from the high density of charged particles in HIPIMS plasma. In particular, it was established that electron-ion Coulomb collisions play an important role on the electron diffusion current to the anode which was considered as being of Bohm type. At the same time, the calculated cathode current is consistent with the experimental discharge parameters.

The use of the OBELIX code for the study of temporal behaviour of a pre-ionized HIMPIS discharge working with a Copper cathode in an Argon gas will be presented and comparison of model data with experimental ones will be made.

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ORAL PRESENTATION

Balance of powers delivered to magnetrons and balance of deposition rates in reactive bipolar pulsed HIPIMS of aluminum oxide

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Dielectric films can be deposited by reactive bipolar pulsed sputtering from two metallic targets. The power balance of both magnetrons is important, as it can influence magnetron lifetimes, film thickness homogeneity and film composition. Unequal power dissipation in both magnetrons can sometimes be observed. Recently, we have shown [1] that the balance of power delivered to both magnetrons in case of reactive sputtering of Al in O_2 depends on the on-times of both magnetrons and on the oxygen partial pressure. The unequal magnetron power dissipation can be compensated by unequal ratio of on-times. Moreover, working in the transition mode resulted in some negative feedback on the power dissipation in case of reactive sputtering of Al in O_2.

Dielectric Al_2O_3 films have been deposited by reactive bipolar pulsed sputtering from two metallic Al targets in Ar + O_2 gas mixture using a MAGPULS BP 1000/100/200 unit. A pair of 2-inch round magnetrons were arranged obliquely in load-lock sputtering system. The substrate holder enabled splitting the sputtered flux of material from each magnetron to one of two substrates. Power input for each magnetron was evaluated from oscilloscopic data of voltage and current.

The bipolar HIPIMS regime used frequency range 1 to 10 kHz, relatively short on-times and high off-times. The peak magnetron current was up to 10 A, that is about 350 W/cm² over the entire target area at about 250 W average power. Voltage control was used to stabilize the reactive working point in the transition mode. The on-time ratio of both magnetrons has been varied bet-ween 75 % and 125 %. The effects on the power balance of both magnetrons are compared with the deposition rates and properties of films, measured mainly by spectroscopic ellipsometry. The films deposited close to 100 % on-time ratio have refractive indices between 1.60 and 1.68 and absorption coefficients below 0.005. Less balanced powers in magnetrons result in different rate of target oxidation of both magnetrons and to modified deposition rates. This also affects the optical properties of films.

Preliminary results of processes depositing Al_2O_3 films by bipolar pulsed reactive sputtering in a larger scale machine equipped with two rectangular magnetrons are also shown.

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ORAL PRESENTATION

Reactive high-power impulse magnetron sputtering of optically transparent zirconium dioxide films

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High-power impulse magnetron sputtering with a pulsed reactive gas flow control, was used for the reactive deposition of optically transparent 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. Two different locations of the oxygen gas inlets above the target with opposite orientations (to the target or to the substrate surface) were used. The repetition frequency of a pulsed dc power supply (HMP 2/1, Huettinger Elektronic) was 500 Hz at the average target power density from 5 Wcm^{-2} to 50 Wcm^{-2} in a period with 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 above the target and their orientation to the substrate surface made it possible to improve quality of the films due to minimized arcing at the sputtered target and to enhance their deposition rates up to 120 nm/min for the target power density of 50 Wcm^{-2} in a period at the duty cycle of 10 %. This is due to benefits of the high-power impulse magnetron sputtering discharges. The zirconium dioxide films were found to be crystalline with a predominant monoclinic structure. Their extinction coefficient was between 1×10^{-4} and 5×10^{-3} (at 550 nm) and hardness between 9 GPa and 16 GPa. Details of the deposition process, including an energy-resolved mass spectrometry at the substrate position (energy distribution functions of positive and negative ions, and compositions of the integral fluxes of positive ions), and measured properties of the films will be presented.

ORAL PRESENTATION

Reactively grown TiN and Ti-Si-N films with high deposition rate using chopped HIPIMS

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Chopped HIPIMS, (c-HIPIMS), a modified version of high power impulse magnetron sputtering (HIPIMS) has been used to deposit titanium nitride and titanium silicon nitride films. Chopped HIPIMS is a variant of HIPIMS,

where a single pulse is decomposed into several individual pulses and tailored through software control in the HIPIMS pulse generator to produce pulse sequences. The films grown by this technique were characterised using XRD, XPS and SEM. Mechanical properties of the films have been assessed using nano-indentation. The deposition rate, when using c-HIPIMS depends upon the choice of c-HIPIMS pulse sequence with systematic changes for both pulse-on and pulse-off times. In particular, deposition rates are higher than for conventional HIPIMS, with an increase of 50 % for TiN growth at comparable parameters, whilst maintaining the dense microstructure typical for HIPIMS. The deposition rates are comparable to direct current magnetron sputtering (DCMS). The increase in deposition rate is ascribed to a combination of reduced gas rarefaction, less self-sputtering and reduced ion trapping. Comparison of the films grown by c-HIPIMS to those when using HIPIMS shows microstructure improvements when using c-HIPIMS. XPS analysis showed that minimal oxygen contents of less than 1 atomic % are found below 10 nm from the top surface, significantly better than for comparable DCMS films. Hysteresis effects and electrical characteristics at the target during the running process have been investigated with various pulse sequences for c-HIPIMS, i.e. varying pulse-on and pulse-off times. The c-HIPIMS films exhibit similar or enhanced properties compared to standard HIPIMS films whilst their deposition rates are increased.

ORAL PRESENTATION

High-power impulse magnetron sputtering of CN_x

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CN_x, referring to under-stoichiometric C_3N_4 coatings, are extensively studied for tribological applications because of their high hardness and low friction coefficient. Nevertheless, it is believed that the best properties would be reached for the ideal βC_3N_4 composition of the coating material. Considering magnetron sputtering-based methods and the need to increase the nitrogen content in the CN_x films, increasing the dissociation and the ionisation rate of the nitrogen molecules in the plasma could be a promising strategy to achieve this goal. Compared to other magnetron techniques used for thin films deposition, HIPIMS (High Power Impulse Magnetron Sputtering) enables a high ionization of the sputtered material as well as a strong dissociation and ionisation of reactive molecules.
In this work, we are aiming at evaluating the relationship between the pulse parameters, the plasma chemistry, and the incorporation of N atoms in the CN_x films.

CN_x films have been synthesized using reactive HIPIMS of a graphite target (450 × 150 mm) in pure N_2 (2.7 10^{-2} mbar). The results are compared to those obtained with a conventional DC reactive magnetron discharge (DCMS) at identical mean power (PD). In HIPIMS, the discharge voltage VD is set to 1400 V and the pulse duration τ is varied between 10 and 40 µs. PD is kept constant at 1 kW by adjusting the frequency accordingly. We discuss the influence of the pulse duration (τ) and therefore of the peak current (I_{peak}) on the chemistry of the film which is determined by X-Ray Photoelectron Spectroscopy (XPS). Optical emission spectroscopy (OES) is performed in order to address the evolution of the plasma chemistry with respect to the increase of the pulse duration.

In HIPIMS, the deposition rate (RD) decreases by increasing I_{peak} and τ. Comparing the HIPIMS data with those recorded for DCMS, it is found that, for τ = 10 µs, RD is slightly higher than in DCMS. For all deposited films, the chemical analysis reveals, a low oxygen content (3-6 % at.) and a N/C ratio close to 0.5 which is slightly higher than the ratio obtained in DCMS. The OES data reveal that, for a given value of PD, the N_2^{+*} emission band intensity is increased as compared to the DCMS discharge likely as a result of increased ionization rate of the N_2 gas.

ORAL PRESENTATION

Deposition of highly insulating aluminium nitride (AlN) by DC and HIPIMS technique: comparison of the two techniques according to plasma investigations and physical analysis

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Using DC reactive magnetron sputtering, good crystalline quality AlN films were deposited on silicon substrate (Rocking curve FWHM around 2 °C) and even epitaxial growth of AlN on AlGaN [1] and ZnO [2] substrates was achieved. These results were obtained thanks to optimization of ion/neutral ratio and the energy of impinging species [3].

In this work, Magnetron DC but also HIPIMS techniques were used for deposition of Aluminium nitride (AlN) thin films. The process was optimized for working with Aluminium target in reactive atmosphere (Ar-N_2). As the plasma characterisation of the ionized species is necessary in order to better understand such a specific plasma process, time-resolved investigations were carried out using Langmuir probe measurements, Optical Emission Spectroscopy and Mass Spectrometry. Physical and chemical properties of the as deposited films are then characterized.

Well crystallised, textured (Rocking curve FWHM around 1°) and thick AlN films were obtained on silicon substrate. Using appropriate substrate for epitaxial growth, some trials have been done by both techniques. The results of such trials will be presented, discussed and a correlation between the films and plasma properties will be made.

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ORAL PRESENTATION

Effect of nitrogen flow rate on the corrosion resistance of ZrN coatings deposited by HIPIMS technology

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Monolayer ZrN coatings were deposited in an industrial scale PVD machine (HTC-1000-4 target system) by utilising HIPIMS technique. Only two cathodes furnished with zirconium targets and with HIPIMS power supplies (Hüttinger Electronic Sp. z o.o., Warsaw, Poland) were operated to deposit the coatings on to 1 micron polished M2 High speed steel (HSS), 304 L Stainless steel (SS) specimens and on Si (100) specimens. Prior to deposition, HIPIMS plasma sustained on a Zirconium (Zr) target was utilised to pretreat the specimens in order to achieve good adhesion. Following pretreatment, coatings were deposited in a mixed N_2 and Ar atmosphere at 400 °C. To analyse the effect of nitrogen flow rate on the stoichiometry and hence on the properties of ZrN coatings, following gas flow ratios (N_2 : Ar) 1 : 2, 1 : 3, 1 : 6 (denoted henceforth as process-P1, P2 and P3 respectively) were employed. All coatings were deposited at -75 V bias with the help of a dedicated bias power supply suitable for HIPIMS (HBP, Hüttinger Electronic) while other deposition parameters were maintained constant.

As anticipated, EDX analysis showed that the effect of N_2 flow rate on stoichiometry of the coatings was very pronounced. The thicknesses of the coatings were found to be 1.88 µm, 1.96 µm and 5.2 µm for the processes P1, P2 and P3 respectively. P1 coatings were found to be over stoichiometric whereas P2 coatings were near stoichiometric and P3 sub-stoichiometric. Coating adhesion was measured by progressive loading scratch tests (CSEM REVETEST) and the critical load (Lc_2) of coating failure was observed under optical microscope. Dry sliding wear rates were measured with a pin on disk apparatus (CSM TRIBOMETER) by sliding a 6 mm Al_2O_3 ball under a load of 5 N at a linear speed of 0.1 ms^{-1} for a total distance of 3500 laps (220 m). Nano hardness values were calculated from the loading-unloading curves obtained from a CSM nanoindentation tester. All coatings exhibited high adhesion (L_{C2} > 100 N), high hardness (in the range of 31-36 GPa) and dry sliding wear coefficient (in the range of KC = 4 – 6 × 10^{-14} $m^3N^{-1}m^{-1}$).

Effect of nitrogen flow rate was also very pronounced on the corrosion resistance of the coatings. The samples were polarised from -1000 mV to +1000 mV in a 3.5 % NaCl solution with the help of a Potentiostat (Gill AC-ACM instruments). Though P1 and P2 coatings were found to have similar E_{Corr} values of 422 mV and 425 mV respectively, in the anodic range of - 320 mV to +100 mV the corrosion current density of P1 (2.9 × 10^{-7} Acm^{-2}) was an order of magnitude higher than P2 (1.6 × 10^{-8} Acm^{-2}). Overall, of all the three coatings, P2 coatings have the lowest current density in the anodic range of -320 mV to +400 mV suggesting a superior corrosion resistance. Even though P3 coatings (sub-stoichiometric) showed the highest (noble) E_{Corr} value of -363 mV they were almost 3 times thicker than the other two coatings. The effect of N_2 flow rate on the microstructure observed by cross-sectional Scanning Electron Microscopy (FEI NOVA-NANOSEM 200) and its influence on the corrosion resistance are discussed.

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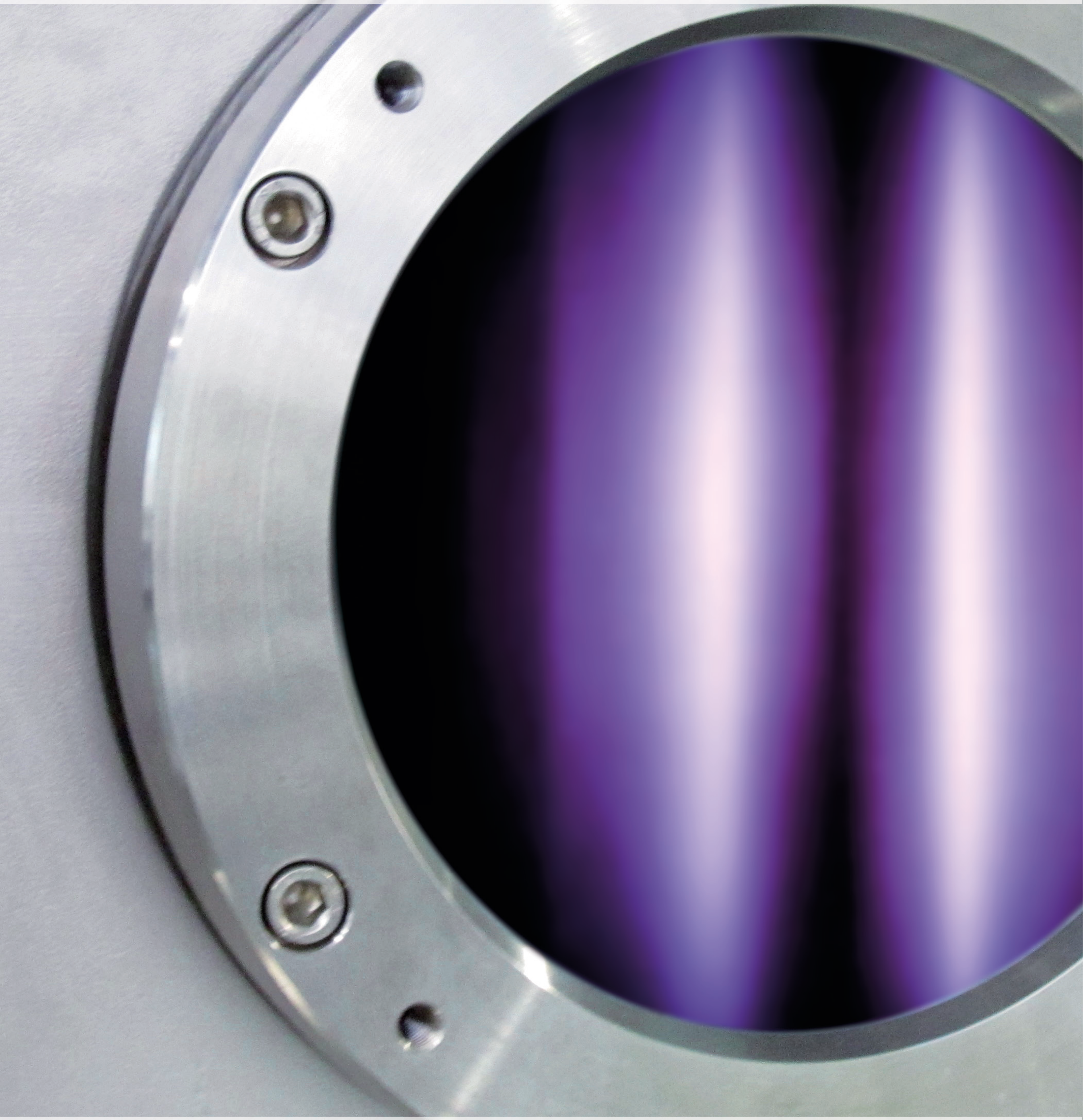

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