HIPIMS 2017

13TH – 14TH JUNE 2017

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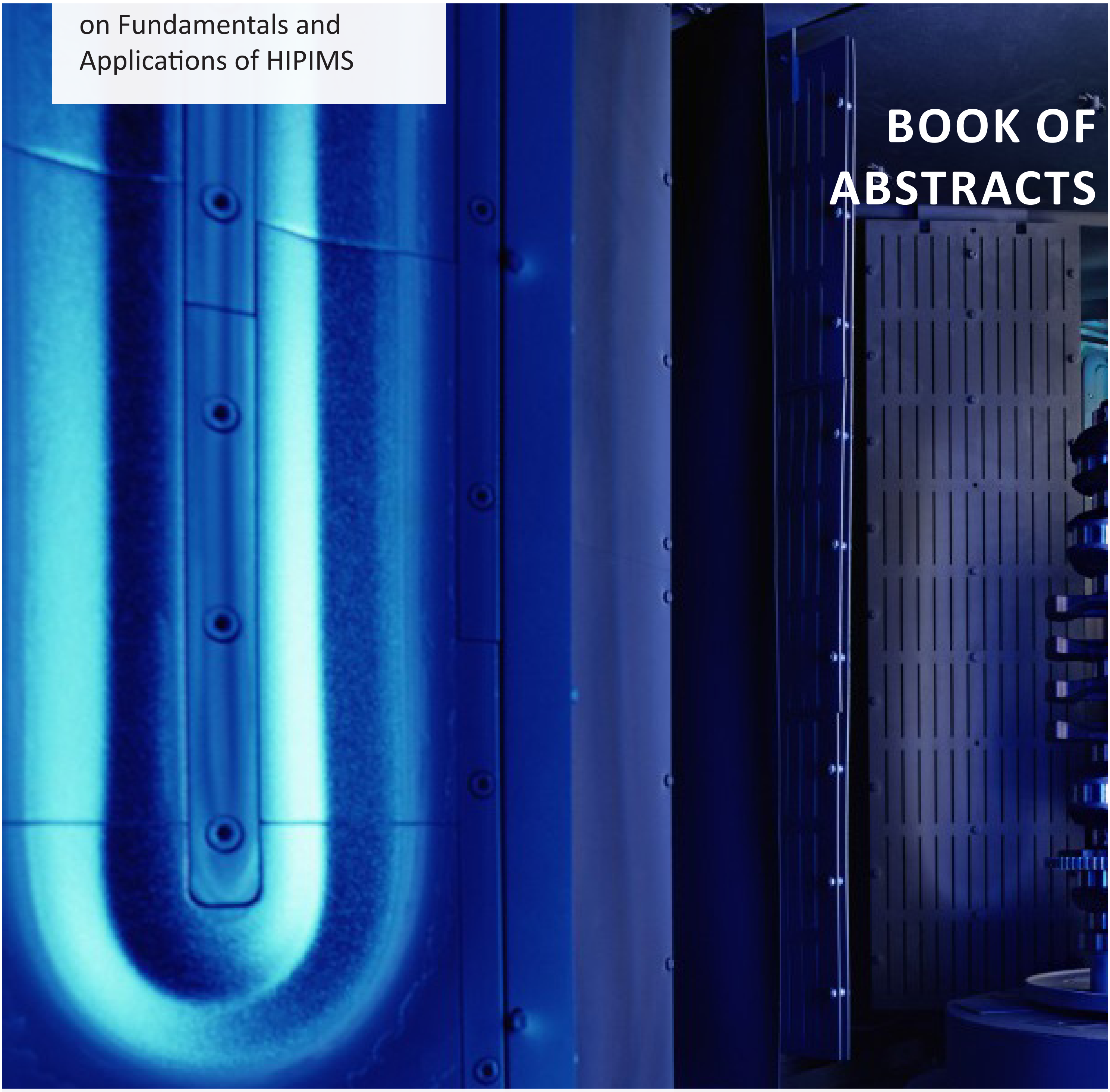
8th International Conference on Fundamentals and Applications of HIPIMS

BOOK OF ABSTRACTS



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Preface

Surface engineering and tailored thin films 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. With the introduction of high power impulse magnetron sputtering (HIPIMS) in 1999, the gap between conventional sputtering and arc evaporation was closed. Since this time, also arc technology strongly benefitted from developments in HIPIMS, especially the development of advanced power supplies and plasma diagnostics suited for high plasma densities.
As well as several international activities the international conference on fundamentals and applications of HIPIMS continues the success story of the HIPIMS days, initiated in 2004 at Sheffield Hallam University, UK. Becoming the only international conference especially dedicated to HIPIMS the HIPIMS conference is a venue for industrial and academic exchange on the latest developments in this fast evolving new technology. As a joint undertaking of Sheffield Hallam University SHU, Network of Competence for Industrial Plasma Surface Technology INPLAS, and Fraunhofer Institute for Surface Engineering and Thin Films IST the HIPIMS conference was launched in 2010 in Sheffield, UK. With approximately 150 delegates the conference is the only topical conference of its size with focus on HIPIMS technology. Indication of the high impact towards applications is the composition of equal shares of participants from research and development (university and research institutes) and industry. Being a global conference representatives from over 25 different countries from all continents usually attend the meetings.
This year's conference focuses on the latest developments in academia and industry. Showing the impact of HIPIMS technology towards industrial applications presentations of industrial applications and products in the field of hard coatings, photovoltaics, optical applications, as well as recent developments in power generation will be presented. Significant increase in activities and high impact is found in the field of reactive HIPIMS and process control. Solutions for reactive process control will be presented. From the material side different results on oxides, carbides and nitrides will be presented. The more academic contributions will focus on fundamental aspects of HIPIMS technology and recent results on modelling.

Dr. Ralf Bandorf and Prof. A. Ehiasarian
Conference Chairman and Co-Chairman of HIPIMS 2017

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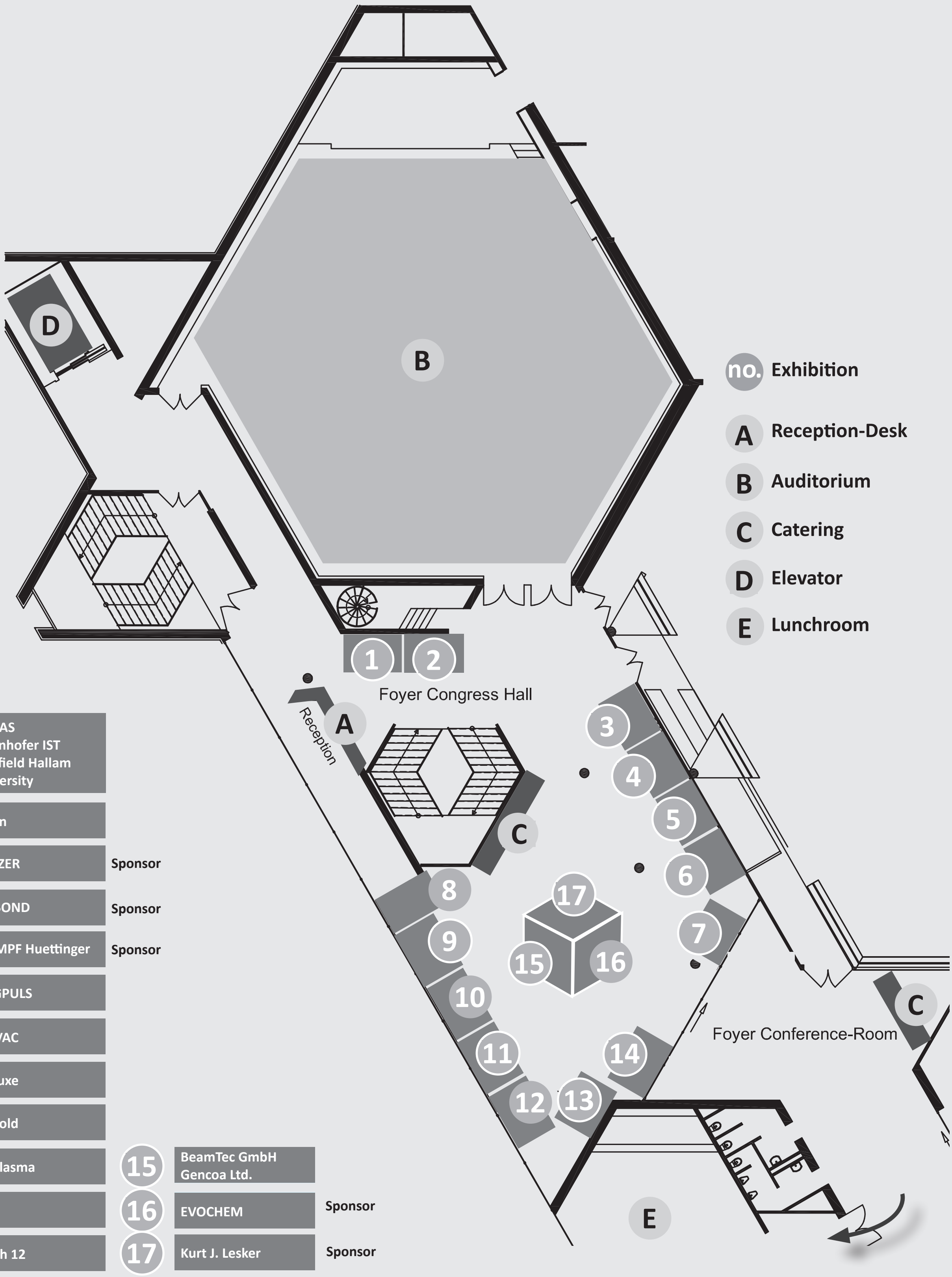

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ORAL PRESENTATION

Abstract 1

Molybdenum Thin Films Deposited by High Power Impulse Magnetron Sputtering for Back Contact Applications

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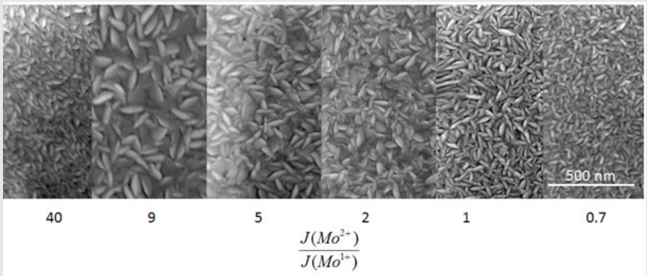
Molybdenum thin films used in chalcopyrite solar cells can influence the Na diffusion rates and the texture of the Cu(InGa)Se2 absorber according to the microstructure and morphology. The lowest resistivity films are achieved at low working pressure and are accompanied by high residual stress and poor adhesion due to the resulting high energy of the deposited flux. High Power Impulse Magnetron Sputtering was employed to ionise the sputtered flux, achieve high adatom mobility at low energy and influence the growth of Mo back contacts. Pulse durations in the range 60 to 1000 μs, sputtering voltages between 800 and 1500 V and deposition pressures of 2×10-3 mbar and 4×10-3 mbar resulted in ten-fold variations in the flux ratios of Mo1+/Mo0 Mo2+/Mo1+, Ar2+/Ar1+ and Mo1+/Ar1+ as determined by optical emission spectroscopy and time-resolved plasma-sampling energy- mass spectroscopy. The energy of metal and gas double- and single-charged ions reduced with pulse duration and increased with voltage. The microstructure of the films varied from open columnar with faceted tops to fully dense as observed by secondary electron microscopy. The removal of double-charged metal ions promoted better defined grain boundaries and a stronger (110) texture as determined from the ratio of Mo I(211) / I(110) diffracted peak intensities and pole figures. The reflectivity of the films improved by 20% compared to industry-standard materials. The lowest resistivity was in the range of 12 μΩ-cm as observed by four-point probe measurements of 570 nm thick films. The correlation between resistivity, microstructure, crystallographic texture and deposition flux characteristics is discussed.



SEM micrograph of the film morphology as a function of the ion flux ratio of Mo2+ and Mo1+.

Abstract 2

On the industrialization of High Power Impulse Plasma Magnetron Sputtering

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High Power Impulse Magnetron Sputtering (HIPIMS) is the youngest Physical Vapor Deposition (PVD) technique available to the industry. The first generation of HIPIMS power supply available commercially was introduced in 2003. For more than one decade the first generation known as Huettinger HMP was successfully applied for deposition of anti-wear and protective coatings, metallization of trenches with high aspect ratio or deposition of transparent insulating as well as conductive oxides, to name just few examples.
Until now anti-wear and protective coating won an established position and are used commercially. Newest market trends show the HIPIMS technology will soon became a standard production also for oxide coatings, both conductive and non-conductive. In order to keep pace with market development the necessary tool, HIPIMS power supply, also requires further evolution to meet high productivity, stability and reproducibility demands of the industry. To fulfill these rigorous requirements new

generation of the HIPIMS TruPlasma Highpulse power supplies offers versatile arc management, unique control of voltage and current peak shape as well as the average power delivery control – sophisticated features previously unavailable in any other HIPIMS power supply units.
In this contribution a comprehensive review of recent improvements of challenging application of HIPIMS technology will be given. First, it will focus on the stability and regulation of the voltage and current pulse shape both in metallic and reactive HIPIMS sputtering. The discussion based on the results on typical metallic targets i.e. Ti and Al will be continued with the analysis of the data collected during sputtering of ITO and GZO (gallium-zinc-oxide) films. The ability to maintain a regulated, stable current level during the pulse and effective arc suppression for continuous operation achievable with new TruPlasma Highpulse units will be analyzed. Finally, the preliminary results will serve to consider advantages and challenges of the HIPIMS application for sputtering of various coatings using single and dual rotatable targets.

Abstract 3

Pure HiPIMS Coatings with 2 µm/hour for Cutting Tool Coatings

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Key feature of this new hardware concept is a deposition rate as high as 2 µm/hour for pure HiPIMS coatings. The paper will present how this equipment does a AlTiN FerroCon® coating in 4 hours 20 mins and a TiAlSiN InoCon® film within 5 hours 20 mins. This data is achieved for 100% HiPIMS mode – not hybrid or mixed set-up –and threefold rotation. An integrated concept of an optimized magnetic set-up of the magnetrons together with the door assembly design of the cathodes –HiPIMS without cable, the pulse unit is right on the chamber door – and a full synchronization between the HiPIMS sources and a dedicated table Bias makes this so far unachieved rate possible.
A scratch load of 120 N for a TiAlSiN coating on a sintered carbide surface indicates enormously high plasma ionization. The dense nature of the films is revealed by nano indentation results showing so far not reachable H^3/E^2 values. SEM images of the fine grain morphology underline this. Machining tests in TiAl6V and in stainless steel show that pure HiPIMS takes the performance of cutting tools to a premium level.

A case study on TiB2 coatings illustrates the benefit of pure HiPIMS coatings: this technology adds to the advantages of sputtering – smooth, droplet free coatings and an unlimited choice of the chemical composition – a tremendously high ionization and hence best adhesion of a dense and uniform coating. The pure HiPIMS technology broadens the application range of TiB2 to cutting tools for highly abrasive workpiece materials.

Abstract 4

Correlation of spatially resolved in-vacuum XPS characterisation and optical diagnostics for magnetron targets in HiPIMS plasma

Layes, V.*[1]; Monje, S.[1]; Corbella, C.[1]; Schulz-von der Gathen, V.[1]; von Keudell, A.; de los Arcos, T.[2]

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In-vacuum characterisation of magnetron targets after high power pulsed magnetron sputtering (HiPIMS) has been performed using X-ray photoelectron spectroscopy (XPS). Al-Cr composite targets (circular, 50 mm diameter) in different geometries were investigated: Al targets, Cr targets, Al targets with a small cylinder of Cr inserted at the racetrack position (composite target) and Cr targets

with a small disk of Al inserted at the racetrack position. The HiPIMS discharge and the target surface composition were characterised for different power conditions. The HiPIMS plasma characterisation was done using optical emission spectroscopy (OES) and fast imaging by a CCD camera; the surface characterisation was done after in-vacuum transfer of the magnetron target to the XPS. This parallel evaluation has provided information about: (i) lateral transport and redeposition of sputtered species on the target, (ii) oxidation state of the target surface as function original composition, position and HiPIMS plasma conditions, and (iii) correlation between local surface conditions and plasma characteristics.

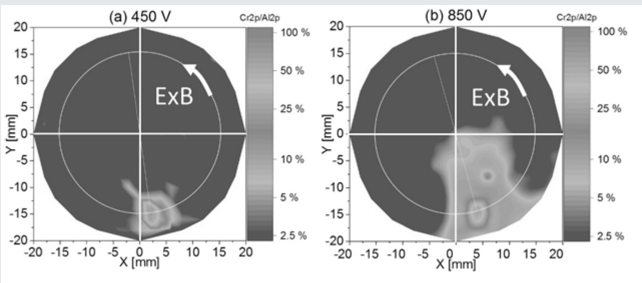

Fig. 1: Chromium concentration map for the low power case (a) and the high power case (b)

Abstract 5

Simulation of heating of the target during High Power Impulse Magnetron Sputtering

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Magnetron functioning during high-power pulse sputtering and also in the mode of “hot cathode” is associated with the target’s heating. It is well known that target’s temperature has a significant impact on the properties of the discharge and deposited coatings. That is why target’s temperature control is being an important and actual task. Direct temperature measurement is a difficult technical problem. It is obvious that in case of using HiPIMS technology temperature is distributed heterogeneously. Simulation is an effective way of temperature definition. The results of simulation of heating the cooled metal target made of different materials (Ti, Ta, Cu) during the high-power magnetron sputtering are presented in this work. It’s peak pulse current and voltage are 1000 A and 1000 V. Target’s diameter is 115 mm, thickness is 5 mm. Erosion profile sizes are: inner diameter is 36 mm, external diameter is 72 mm. One of the target’s sides is cooled by water and has constant temperature 283 K. Heat is carried by radiation and thermal conductivity. Simulation was conducted in the program COMSOL Multiphysics by solving a differential equation of thermal conductivity using a method of finite elements. Power pulse was simulated by using measured waveforms of current and voltage. As a result it is shown that during high-power pulse magnetron sputtering metal target is being heated up to the melting temperature. Parameters which have the most influence on the temperature are: pulse frequency, pulse width, duty cycle and energy. Also temperature pulsations during impulses influence were detected.

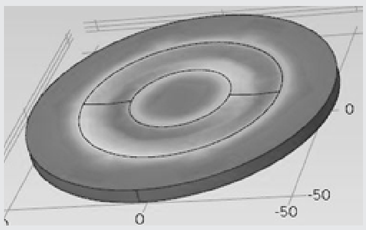

Fig. 1: The result of simulation of impact of pulses on titanium target on the tenth second (duration 250 µs, frequency 100 Hz).

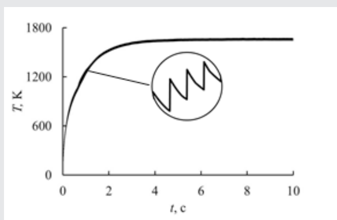

Fig. 2. Dependence of maximum temperature of titanium target of time (duration 250 µs, frequency 100 Hz); also additionally were shown pulsations between high-power pulses.

Abstract 6

Study of spoke rotation, merging and splitting in HiPIMS plasma

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Study of self-organized structures called spokes was performed in the HiPIMS plasma using simultaneous broadband optical screening via ICCD camera (200 ns time scale) and the embedded probes measuring the local current delivered by the spoke to the target. As a spoke passed over a set of embedded probes in the niobium cathode target, a distinct local current modulation is observed. Typically the current modulation was up to twice the average value, matching well with the radially integrated optical emission intensities obtained by the ICCD.
The dual diagnostic system enabled the observation of a set of spokes as they rotated and the events of the spoke merging and splitting were recorded. The two spokes with similar sizes and intensities were observed to merge into one larger spoke, while the retaining the velocity of the trailing spoke. In the merged spoke both the plasma emission intensity and current collected by the embedded probes was redistributed to have their maximum at a trailing edge. The reverse process, in which spokes split was also observed. The total charge collected by the embedded probes during the spoke splitting was conserved. After the spoke merging or splitting events occurred, the new spoke configuration was not always stable in time. Often the large spoke split into two smaller spokes only to reform a short time later. However for a given experimental conditions only a slight variation from the average mode number m was observed (typically a change of $\Delta m = 1$). In addition a simple phenomenological model was developed to relate the spoke mode number m with the spoke dimensions, spoke velocity and gas atom velocity.

Abstract 7

Potential Structure of Ionization Zones and Implications for Electron Heating and Ionization Dynamics

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In most (but not all) situations, HiPIMS discharges exhibit “spokes”, i.e. zones of enhanced excitation and ionization that move along the magnetron’s “race track” [1-3]. Electrons drift in the E x B direction with a drift velocity about one order of magnitude faster than the motion of ionization zones and they may encounter ionization zones more than one time before arriving at the anode or other place. Recent research confirmed that those zones are locations of higher plasma potential relative to the potential of the surrounding regions [4]. There is a rather sharp transition from low to high potential on the side where electrons arrive. This implies that electrons moving from low to high potential at this location and gain energy. Emissive probe measurements for the relatively simple case of dc magnetron sputtering showed that the potential jump is of order 50 V or higher, i.e., clearly exceeding the ionization energies of process gases and sputtered atoms [4]. The existence of a spoke-centered potential hump and related electron energization was suspected in an earlier work [5] based on ion energy measurements. This can be considered confirmed by emissive probe measurements. However, emissive probe measurements are conclusive for dc magnetron sputtering in the presence of a “regular”, self-organized spoke patterns but much more difficult to do in the HiPIMS case where we have evidence for turbulence and chaotic behavior [6], as manifested by merging and splitting spokes, and emission of plasma flares

[7]. In this presentation, electron heating in the spoke's potential structure is emphasized and compared with the conventional heating described by Thornton's paradigm: electron heating by secondary electrons which are energized in the target's sheath. It is argued that localized electron heating and the appearance of spokes are mutually related (not unlike the "chicken and egg" problem).

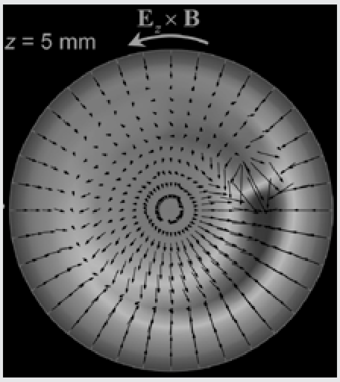

Figure: Plasma potential measured for the case of one dcMS spoke, red: positive and blue: negative, with the largest difference about 80 V, for more details see [4].

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[7] P.A. Ni, C. Hornschuch, M. Panjan, A. Anders, Appl. Phys. Lett. 101 (2012) 224102.

Abstract 8

Synchronised external magnetic fields applied in HiPIMS enhance plasma generation and plasma transport

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Over the last decade High Power Impulse Magnetron Sputtering (HiPIMS) has been shown to be an excellent technique for depositing dense, high quality thin films in both non-reactive and reactive modes. The high degree of ionisation of the depositing species allows fine-tuning of film properties and microstructures by the simple application of substrate bias. The main disadvantage of HiPIMS that has limited its translation to many commercial applications is its low deposition rate. Magnetic field guiding of the plasma from the racetrack where it is produced to the substrate where it participates in thin film deposition has been suggested as a way of enhancing the deposition rate. In this presentation, we explore the use of externally applied pulsed and steady magnetic fields for the enhancement of deposition rate in both reactive and non-reactive HiPIMS. External magnetic fields were applied by a solenoidal coil that was placed above the magnetron target. When the solenoid field was parallel to that of the central magnetron magnet the HiPIMS plasma became unstable, whilst when excited to produce a field in the anti-parallel direction, the plasma became denser and its transport to the substrate was enhanced, improving the deposition rate. This behaviour can be explained by calculations of the total magnetic field, which show a strengthening of fields above the racetrack in one case and a weakening of these fields in the other.

In the case of a steady magnetic field, a higher voltage was required to initiate the HiPIMS discharge, a longer

delay time was observed for current onset, and oxide films deposited in reactive HiPIMS became substoichiometric. In contrast, for the pulsed magnetic field, film stoichiometry was maintained under all applied external magnetic field strengths. Varying the duration and delay times of the magnetic field after the application of the HiPIMS voltage pulse revealed that the afterglow of the plasma between HiPIMS pulses was actively quenched by the presence of the magnetic field. Therefore, the optimum operation with the highest plasma density was obtained by applying the external magnetic field only when the plasma was established and removing it at the end of the HiPIMS pulse.
A model to explain the findings is presented. We describe an approach to achieve maximum deposition rate while maintaining film stoichiometry and high film quality. Amorphous HfO2 films with leakage current through the film of less than 5 × 10−5 A/cm2 at 0.1 MV/cm were obtained at the maximum deposition rate. The refractive index, at a wavelength of 500 nm, of the film prepared with pulsed magnetic field was 2.05 with a very low extinction coefficient of 8 × 10−5.

Abstract 9

Effects of Bipolar Pulses in High Powered Impulse Magnetron Sputtering (HiPIMS)

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High Power Impulse Magnetron Sputtering (HiPIMS) has been proven as an ionized physical vapor deposition (iPVD) technique to provide superior film quality and adhesion. The ionization of the target material is done without the use of additional plasma generators through high power densities at the target surface, ultimately increasing the ion flux to the substrate. The incident ions allow for controlled ion bombardment energy leading to superior quality films over that of direct current magnetron sputtering (DCMS). One limiting factor of implementation of HiPIMS in industrial settings is the decreased deposition rates as compared to conventional DCMS. The reduction in deposition rate is believed to be mainly due to the return of ionized target material back to the target surface. The Center for Plasma-Material Interactions is working with a new bipolar HiPIMS power supply module for use in experimental process development. The bipolar supply allows for control, both potential and timing, of a positive polarity pulse following the negative polarity main pulse. Timed control of the substrate bias with respect to the HiPIMS pulse, was proven to provide control of film growth morphology and reductions in the film stresses [1]. Previous works have reported increases in deposition rates through manipulation of the pulse waveform by creating multiple pulses within the full HiPIMS pulses, namely “chopped” HiPIMS (c-HiPIMS). It is believed that allowing the working gas to replenish, rarefaction was limited and therefore deposition rate increases of up to 50% over standard HiPIMS with a Titanium target were recorded [2]. Another method previously investigated by the University of Illinois at Urbana-Champaign to increase the deposition rate of HiPIMS is the TriPack magnet pack [3] is explained by a modified transport model showing a reduction in return effect. Increased deposition rates were achieved by extending the ionization region away from the target surface while maintaining high ionization probabilities [4]. This work investigates the use of a controlled positive polarity pulse and increased ionization regions to increase deposition rate while controlling ion bombardment energy without an external substrate bias, retaining the superior film qualities expected from HiPIMS. This paper includes investigations of deposition rate, ion-neutral fraction and currents, and film qualities.

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Abstract 10

Improve the Coating Properties by Modeling and Optimizing the HiPIMS Magnetron Configuration

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According to related reports and experiments, we can know the magnetic field distribution of the magnetron plays an important role in the HIPIMS technology. For example, the discharge ionization rate, sputtering and deposition rate are extremely sensitive to small change of magnetron field distribution. From the cross-knowledge of magnetron sputtering and electromagnetic field theory, (1) the reasonable magnetron distribution and intensity can capture the electrons of the plasma, restrain and extend the electron trajectory, then improve the ionization rate effectively. (2) Under the action of quadratic electromagnetic field, the drift motion of secondary electrons mainly occurs in the area where the magnetic field lines are perpendicular to the electric field lines. That is the position of the maximum horizontal component of magnetic field Br corresponds to the deepest point of the erosion trajectory. So the field distribution of the magnetron source has also a fundamental effect on the target erosion
Therefore, finite element method is used to calculate and model the magnetic field of magnetron in this paper. The influence of different magnetron distributions on the erosion trajectory is analyzed by a lot of modeling. In order to improve the target utilization, we try to design a new configuration to increase the distribution of the magnetic horizontal component above the target surface. Moreover, the modeling can be used to predict the influence of magnetic field distribution and parameters on the film properties and provide some physical information that is difficult to observe from experiments.

Abstract 11

Unrevealed process enhancements by engaging an Active Positive Voltage reversal in HiPIMS applications

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This paper demonstrates the effect of an active, optimized short positive voltage reversal, right after the negative HIPIMS pulse. It reveals the technological benefits on the plasma discharge characteristics and coating properties.
The instant advantage is that the magnetron surface will be immediately discharged, which will reduce the tendency to arcing. However, there are several other effects observed during the performance of reactive sputtering, such as enhanced high energetic positive ion bombardment towards the substrate. Due to the raise of the plasma potential, higher incorporation of reactive species into the depositing film, enhanced deposition rates and elastic hardness values are observed.
Data on the reactive process evolution using Optical Emission Spectroscopy, deposition growth rates, coating microstructure, composition and hardness are presented for both Titanium Nitride (TiN) and Tantalum Nitride (TaN). It is shown that the deposition rates as well as the coating hardness can be enhanced up to 25%.
The described voltage reversal feature, the hiPlus, has been implemented and is available as an option in the hiP-V HiPIMS power supply product line.

Abstract 12

Improving deposition rate of titanium nitride-based films using a superimposed high-power impulse and middle-frequency magnetron sputtering technique

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The low deposition rate in high power impulse magnetron sputtering (HiPIMS) somewhat limits its commercial applications in thin film and coating technologies. The major cause often reported is the high degree of ionization that takes place in the plasma discharge and this may lead to a self-sputtering phenomenon, which further downgrades the sputtering efficiency. It is somehow contradictive, because on one hand the high ionization degree in HiPIMS improves the film density and properties but on the other hand the low deposition rate is problematic. In this work, the ion properties in HiPIMS are investigated and compared to its direct current (DC) counterpart in the case of titanium nitride-based sputter depositions. A plasma sampling energy-resolved mass spectrometer was used to measure the ion energy distribution function (IEDF) of metal and gas ions. After understanding the properties of ions in the plasma, we offer an effective way to improve the deposition rate by introducing the middle-frequency (MF) pulses during off-time of HiPIMS pulsing. It is found that the deposition rate has been improved from 4.4 to 29.3 nm/min without significantly deteriorating the mechanical properties and adhesion quality of deposited films. The mechanism of deposition rate improvement is thus proposed and discussed.

Abstract 13

An ionization region model ofthe reactive Ar/O2 high power impulse magnetron sputtering discharge

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In the reactive high power impulse magnetron sputtering discharge experimental findings indicate that there is a significant increase of the discharge current [1]. A reactive ionization region model (R-IRM) is developed to describe the reactive Ar/O2 high power impulse magnetron sputtering (HiPIMS) discharge with titanium target. It is then applied to study the temporal behavior of the discharge plasma parameters such as electron density, the neutral and ion composition, the ionization fraction of the sputtered vapor and the oxygen dissociation fraction. We study and compare the discharge properties when the discharge is operated in the two well established operating modes, the metal mode and the poisoned mode. Experimentally, it is found that in the metal mode the discharge current waveform displays a typical non-reactive evolution, while in the poisoned mode the discharge current waveform becomes distinctly triangular and the current increases significantly [2]. Using the R-IRM we find that when the discharge is operated in the metal mode Ar+ and Ti+ -ions contribute most significantly (roughly equal amounts) to the discharge current while in the

poisoned mode the Ar+-ions contribute most significantly to the discharge current while the contribution of O+ -ions and secondary electron emission is much smaller [3]. Furthermore, we find that recycling of ionized atoms coming from the target are reqired for the current generation in both modes of operation. In the metal mode self-sputter recycling dominates and in the poisoned mode working gas recycling dominates, and it is concluded that the dominating type of recycling determines the discharge current waveform. The density of atomic oxygen increases significantly as we move from the metal mode to the transition mode, and finally into the compound (poisoned) mode. The main gain rate responsible for the increase is sputtering of atomic oxygen from the oxidized target [4]. Both in the poisoned mode and in the transition mode, sputtering makes up more than 80 % of the total gain rate for atomic oxygen.

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Abstract 14

The role of metal implantation in reactive high power impulse magnetron sputtering

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Many of technologically important dielectric oxide films are prepared using reactive magnetron sputtering from metal target in a reactive gas atmosphere. Reactive high-power impulse magnetron sputtering (HiPIMS) has recently been used for preparation of various optically transparent non-conductive metal oxides, such as TiO2, ZrO2, Ta2O5, HfO2, optically transparent conductive oxides, such as InSnO, Al-doped ZnO, or thermochromic VO2 films. Improved film properties compared to films prepared by dc magnetron sputtering were reported.
To improve the understanding of the complicated processes in the reactive HiPIMS discharges, we have developed a time-dependent parametric model of the deposition process [1]. The model takes into account specific features of HiPIMS discharges, namely, gas rarefaction and ionization in front of the sputtered target, backward flux of the ionized sputtered metal atoms and reactive gas atoms onto the target, and high degree of dissociation of reactive gas molecules in the fluxes onto the target and substrate. We have found that the return of metal ions decreases the compound fraction in the target surface layer. However, this process has been somewhat simplified in up-to-date reactive HiPIMS models and, therefore, we believe that the role of the returning ions has to be studied in more detail. We present an extension of the parametric model that features a depth-resolved target composition (with several equidistant depth layers compared to two layers in the original model) and a volume balance equation for the reactive gas. The model is solved for several discharge conditions based on experimental data obtained during reactive sputtering of Al and Ti in Ar + O2 atmosphere. Sputtering yields and implantation profiles of reactive gas and metal atoms calculated by a binary-collision approximation Monte Carlo method using the SDTrimSP program were used in the simulations. In the discussion, we focus on the effect of return and implantation of metal ions on the composition of the target.

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Abstract 15

r-HiPIMS of magnesium oxide

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While the mechanisms that drive the metallic HiPIMS discharges are well studied, the influence of a reactive gas addition to the plasma is still under investigation. Indeed, a limited number of experimental results on reactive HiPIMS (r-HIPIMS) can be found in literature. This hinders to answer some fundamental questions of which the presence of a hysteresis in the deposition parameters sticks out like a sore thumb.
The large difference in deposition rate between metallic and poisoned mode during DC magnetron reactive sputtering of magnesium in a Ar/O2 atmosphere results in a distinctive hysteresis. Moreover, the transition between the two modes can easily be traced by the discharge voltage changes as the secondary electron yield ratio between MgO and Mg is large. This makes the Mg/Ar/O2 system the ideal candidate to investigate the presence of hysteresis in reactive HIPIMS.
In this paper the discharge voltage hysteresis is first studied in the “classical” way by stepwise increasing and decreasing the oxygen flow. Also an alternative method is applied by sweeping the discharge voltage at a fixed oxygen flow. In this way, the transition region between metallic and poisoned mode can be accessed. The influence of duty cycle, frequency and pressure on these two types of experiments as well as the impact of target poisoning on thin film growth will be discussed.

Abstract 16

High-rate reactive HiPIMS deposition of Hf-O-N films with smoothly controlled composition

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Oxynitrides are a class of materials with yet unexplored physical, chemical and functional properties, and a great potential for industrial applications.

In this work, reactive HiPIMS with a feedback pulsed reactive gas (oxygen and nitrogen) flow control and an optimized location (high-density plasma) of the reactive gas inlets in front of the target and their orientation toward the substrate made it possible to produce high-quality Hf-O-N films with a tunable elemental composition, structure and properties at very high deposition rates ranging from 240 nm/min for HfO2 films [1] to 175 nm/min for HfN films. Basic principles of this method, maximizing the degree of dissociation of both O2 and N2 molecules in a discharge plasma, is given.

The depositions were performed using a strongly unbalanced magnetron with a planar hafnium target of 100 mm diameter in argon-oxygen-nitrogen gas mixtures at the argon pressure of 2 Pa. The nitrogen fractions in the reactive gas flow were in the range from 0 % to 100 %. The repetition frequency was 500 Hz at a fixed deposition-averaged target power density of 30 Wcm-2 with the voltage pulse duration of 200 μs (duty cycle of 10 %). The substrate temperatures were less than 140 °C during the depositions of films on a floating substrate at the distance of 100 mm from the target. All films were nanocrystalline and their elemental compositions were varied gradually from Hf32O66 to Hf57O6N33. We present a gradual change of hard (18 GPa), highly optically transparent

(extinction coefficient of 5x10-4 at 550 nm), electrically insulating and hydrophobic (water droplet contact angle of 101°) HfO2 films into harder (25 GPa), optically non-transparent, electrically conductive (electrical resistivity of 3.2x10-6 Ωm) and more hydrophobic (water droplet contact angle of 107°) HfN films [2]. Particular attention is paid to Hf-O-N films with a low N content (≤ 4 at.%). The results are important for designing oxynitride coatings, and pathways for their preparation, for various technological applications.

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Abstract 17

The target condition dependent optical and electronic functionalities of WO3 and WOxNy films deposited by reactive HiPIMS

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In pulsed reactive sputtering, the compound layer on the target surface (‚poisoning‘) is eroded during the pulse, and the compound layer forms during the time in-between the pulses. The cycle of erosion and formation of compound layer during pulse cycles in reactive HiPIMS opens up the possibility of tuning discharge conditions and the functional properties of deposited films through altering the duty cycle, while without changing the reactive gas mixture.
Two different reactive systems; tungsten in oxygen, and tungsten in oxygen/nitrogen were studied, in which amorphous films of tungsten oxide (WO3) and tungsten oxynitride (WOxNy) were deposited at room temperature.
The evolution of chemical changes in the target surface depends upon the physicochemical properties of the target material such as the affinity of the target for the reactive gas mix and the compound layer melting point and volatility.
The pulse length induced modification of the compound layer on tungsten target and the consequent modulation of discharge chemistry and properties of the deposited films had been investigated. The variations in optical properties of different layers of WO3 and WOxNy films deposited for different HiPIMS pulse durations were investigated by spectroscopic ellipsometry, using the Arwin and Aspnes method of fitting, which is a highly effective tool to monitor the grading optical characteristics in real time. With the increase in duration of discharge pulses, the refractive index of the WO3 films were progressively reduced, whilst that of the WOxNy increased. Howeve, variations in the leakage current through the films was observed only for WOxNy films. These variations in optical and electrical properties of the films could be attributed to changes in chemical composition and/or densification of depositing films.
Tuning the pulse characteristics holds great promise for the fabrication of multilayer films with graded optical and electrical functionalities.

Abstract 18

HIPIMS Peak Power to Affect Film Adhesion of Titanium and Titanium Oxide Films on PET Substrate

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High power impulse magnetron sputtering (HIPIMS), offering high density, high ionization plasma and low temperature capability, has opened a possible gateway for producing functional thin films on flexible and even stretchable substrate. The peak power of the HIPIMS power supply, known to garven film properties, has yet fully revealed its effect on the film adhesion strength. In this study, metal titanium (HIPIMS-Ti) and titanium oxide (HIPIMS-TiO2) was deposited separately on flexible polyethylene terephthalate (PET) substrates. Peak power was adjusted by controlling pulse width (Ton) and pulse frequency (f) to reveal the effect on the film adhesion. Microstructure and mechanical properties, film adhesion in particular, of the prepared films were revealed to clarify their relevance.
Experimental results show that high peak power level of 0.6 kW/cm2 and 1.0 kW/cm2 for both HIPIMS-Ti and HIPIMS-TiO2, separately can be achieved when operated at both low pulsed width and pulse frequency. The OES spectra show that when at higher peak power level, both HIPIMS-Ti and HIPIMS-TiO2 result in an increase in the amount of titanium ions in the plasma space associated with a decrease in deposition rates for both HIPIMS-Ti and HIPIMS-TiO2 films. A typical HIPIMS featureless topography can be obtained with its film adhesion on PET substrate ultimately reached. This enables the HIPIMS-Ti film to remain adhered strongly until the PET stretched to fracture. In the case of HIPIMS-TiO2, it presents a relatively week film adhesion on PET and a metal interlayer can improve this.

Abstract 19

MEASUREMENTS ON A HIGH VOLTAGE PULSED SUBSTRATE (PBII) IN A HIPIMS PROCESS

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In a novel experiment a HiPIMS discharge was combined with plasma based ion implantation (PBII). Proper synchronization of the HiPIMS and PBII pulses allows successive and simultaneous coating and doping of the substrate surface in a complex, finely adjustable system. The delay between the HiPIMS and the PBII pulse is a critical parameter for the synchronization of the pulses. To investigate the effect of this parameter on the energy flux towards the PBII substrate, VI-probe and calorimetric measurements were performed.
The energy flux was measured utilizing a specially designed setup for indirect calorimetric measurement of the high voltage pulsed substrate. The results reveal the effect of the delay on the energy flux and ion current to the substrate for different PBII pulse durations and PBII voltages. A maximum of electrical power and energy flux was found for delay values significantly longer than the duration of the HiPIMS pulse. This maximum is explained to be caused by an ion wave/bunch which originates at the target and travels towards the substrate with the energy obtained from the sputter process. The investigation of different PBII pulse durations and PBII voltages revealed that these parameters only affect the absolute values of our measurements but do not influence the arrival time of the ion wave.

Abstract 20

HiPIMS coatings for SRF cavities: influence of process parameters on film morphology and cavities performances.

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Superconducting accelerating radiofrequency (SRF) cavities are the components used in particle accelerators to provide energy to charged particles. Over the last 30 years CERN has developed an expertise in thin film based SRF cavities using copper cavities coated with niobium layer [1]. Such an approach allows the reduction of the fabrication costs and suppresses the possibility of having a quench event with respect to their counterparts made out of bulk niobium thanks to copper thermal conductivity.
The standard coating technique being used is magnetron sputtering as it provides high coating rates using ultra-pure source material and thus reducing the probability of film contamination that could lead to poor superconducting performances.
However it has been shown that this technique exhibits some limitations especially concerning the film density directly linked to the impinging angle of the niobium atoms onto the cavity surface during the sputtering process [2, 3]. Such film features can successfully be predicted using a monte-carlo simulation and are experimentally confirmed in this work by microscopy imaging.
The porosities observed are one candidate to explain the so-called Q-slope effect observed in this type of cavities which results in an exponential increase of the RF surface resistance of the Nb film when increasing the RF field within the cavity.
In order to address such limitations it has been proposed to use HiPIMS coating instead of DCMS to go toward a densification of the layer.
We will first show that biased HiPIMS leads to significantly improved film density all over the cavity surface when compared to DCMS coatings based on focused ion beam investigation on samples. The effect of the coating parameters on the surface resistance and cavities performances will also be discussed. Finally we will investigate the possibility of using an unbalanced plasma source to tune the coating profile with the perspective of coating even more complex cavities' shapes.

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[3] D. Tonini, MORPHOLOGY OF NIOBIUM FILMS SPUTTERED AT DIFFERENT TARGET-SUBSTRATE ANGLE, SRF 2003.

Abstract 21

HIPIMS deposited CrN/NbN coatings to preserve the mechanical properties of the substrate material and protect against steam oxidation and water droplet erosion attacks

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A significant reduction of CO2 emissions is expected by increasing the efficiencies of the steam turbines to η > 50% which can be achieved by moving from subcritical low pressure/ low temperatures, to high pressure/ high temperature, ultra-supercritical regime of operation. The main challenges faced by different steel components of the power plant with this approach however, consist of material failure due to high temperature oxidation, and phenomenon such as creep, erosion and descaling after a stipulated period of time. In the current work, 4 µm thick CrN/NbN coating utilising nanoscale multilayer structure with bi-layer thickness of Δ = 3.4 nm has been used to protect low Cr content P92 steel widely used in steam power plants. The novel High Power Impulse Magnetron Sputtering (HIPIMS) deposition technology has been used to deposit CrN/NbN with enhanced adhesion (critical scratch adhesion value of Lc= 80N) and very dense microstructure as demonstrated by XTEM imaging. P92 coated samples were oxidised at 600°C in 100% high pressure, 50 bar steam atmosphere up to 1500 h. The gas-flow velocity through the reaction zone of the test rig was 0.0133 m/s. In these conditions CrN/ NbN provided reliable protection of the P92 steel. This research also revealed that unlike other state-of -the-art PVD technologies, HIPIMS does not have an adverse effect on the mechanical properties of the substrate material, which is of paramount importance in case of turbine blade applications. In high temperature (650oC) tensile strength test uncoated P92 steel showed Ultimate Tensile Strength (UTS) values of 229 MPa and Yield Strength, (YS) values of 222 MPa compared to UTS = 307 MPa and YS = 291 MPa measured for CrN/NbN coated P92 steel. Similarly in strain controlled, (0.4% strain) Low Cycle Fatigue tests at 650oC both uncoated and coated specimens failed after similar number of cycles, Nf = 1700 and Nf= 1712 respectively and showed similar half-life stress drop of -37% and -43% respectively. Finally high temperature creep tests at 650oC, tensile stress of 120 MPa revealed that the HIPIMS coating improved the creep lifetime by almost factor of two from 564 hours to 908 hours whereas the creep rate was decreased from 17.6 10-6 s-1 to 13.5 10-6 s-1. The protection properties of the coating against water droplet erosion attack were tested using specialized test rig. The coating shows high resistance against water droplet erosion. After 2.4E6 impacts no measurable weight loss was detected.

Abstract 22

Phenomenological study of the influence of HiPIMS process parameters on the tribomechanical properties of TiAlN coatings

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High Power Impulse Magnetron Sputtering (HiPIMS) has demonstrated to have great potential for industrial applications. The utilisation of high power density in short pulses results in the creation of high-density plasma and in the high ionisation of the gas and sputtered species. Investigations on this new PVD technique has increased in comparison with traditional magnetron sputtering due to the possibility to obtain dense, homogeneous and tougher coatings with an increased adhesion to the substrates. TiAlN PVD coatings have been deposited by different sputtering methods such as arc ion plating, direct current magnetron sputtering and plasma assisted chemical vapour deposition due to its high hardness, wear resistance and relatively high thermal stability. In the present work, TiAlN coatings are deposited using HiPIMS technology to investigate the influence of pulse frequency and pulse duration (in terms of duty cycle) on the resulting TiAlN HiPIMS coatings. During the deposition, the pulse frequency and pulse duration were varied from 500 to 1000 Hz and 200 to 50 µs respectively. The characterisation of the microstructure of TiAlN HiPIMS coatings was performed by SEM and their chemical composition evaluated by EDX and GDOES. Additionally, phase analysis is performed by means of XRD in order to determine the crystalline compounds in the coatings and evaluate their occurrence and relative amount with different deposition conditions. Complementary to the coating structural characterisation, nanoindentation, scratch test, Rockwell C adhesion test and pin on disk test were performed and evaluated.

Abstract 23

Adhesion enhancement of DLC hard coatings by HiPIMS metal etching: a comparison between Titanium and Chromium

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Diamond-like carbon (DLC) coatings have been recognized as one of the most valuable engineering materials for tribological applications. Their excellent frictional behavior combined with high surface hardness, offers an extraordinary protection against abrasive wear. These properties suit the requirements to prolong tool life in machining applications. Nevertheless, the adhesion of DLC coatings to the steel substrates is still a key factor determining their performance and successful implementation in industrial production. In this work, we present a process to improve the adhesion properties of the coating using HiPIMS technique. Highly ionized metal flux generated with HiPIMS allows to efficiently remove contaminants of the surface and to incorporate metals into the substrate. A comparison with chromium and titanium pre-treatment was carried out to evaluate the influence of factors such as the degree of ionization, the energy flux or the depth of incorporation. Microstructural modifications are observed in the coating-substrate interface with HRTEM and the depth of implantation is evaluated with EELS spectroscopy. A crystallographic alignment from substrate to coating is obtained for higher energetic pretreatment processes. This factor together with the chemical affinity contributes in a decisive way to improve the adhesion. Adhesion enhancement is examined with Rockwell, nanoscratch and scratch testing, obtaining outstanding critical load values for those coatings prepared with HiPIMS pretreatment method.

Abstract 24

HIPIMS-Arc carbon films on 500 mm cathodes

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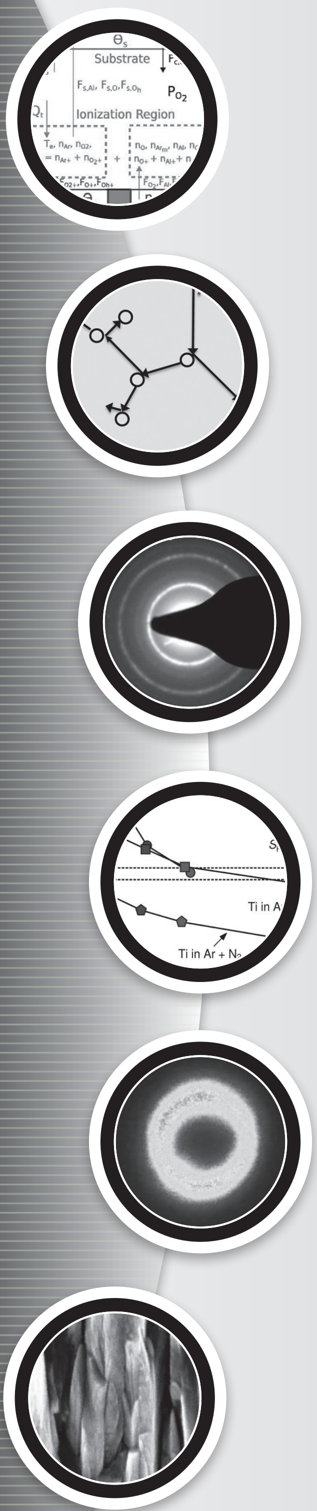
Since the early years of HIPIMS evaluation, the deposition of hard carbon or diamond-like carbon films was of high interest. Some publications on high density but low hardness or only very thin films deposited with e.g. neon instead of argon exist. Introducing a HIPIMS to Arc mixed mode by M. Lattemann et al opened the scene for combining the high density sputtering of smooth coatings with the high energy processing by arc. In 2015 Ganesan et al. reported on the deposition of ta-C films with high sp3-content from a 3 inch target. This work focusses on the realization of a HIPIMS-Arc mixed mode process on an industrial size cathode to bridge the gap between R&D and industry. Process parameters and basic aspects on how to design a pulse for transitioning into an arc will be discussed. The plasma was characterized by optical emission spectroscopy. OES offers clear indication on the generation of carbon ions that can be used for tailoring the process. Finally, the Vickers hardness of the prepared films was measured.


Substrate
Ionization Region
Ti in Ar + N2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Poster presentation

Abstract 25

A controlled high-rate reactive HiPIMS deposition of ZrO2 films: an optical emission spectroscopy study

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Effective deposition of dielectric oxide or/and nitride films by a high-power impulse magnetron sputtering (HiPIMS) is a challenging task. At our department, a feed-back pulsed reactive gas flow control (RGFC) system had been developed to utilize exclusive benefits of the HiPIMS in a high-rate reactive deposition of stoichiometric oxide or/and nitride films [1, 2].
Here, we report on the results of the optical emission spectroscopy with a temporal resolution of 320 ns carried out near the target and in the plasma bulk during the deposition of densified ZrO2 films using the HiPIMS controlled by the pulsed RGFC system. Depositions were performed in a stainless-steel vacuum chamber equipped with an unbalanced magnetron (Zr target, the diameter of 100 mm). Oxygen was admitted directly into a discharge plasma via two corundum conduits (located at 25 mm from the target) with the inlets (diameter of 1 mm) directed to the substrate. The actual O2 flow rate through the conduits was adjusted by the pulsed RGFC system during the depositions according to the monitored value of the average discharge current in a period of the power supply that oscillates in compliance with the instantaneous local O2 partial pressure in the discharge. The repetition frequency of voltage pulses with a duration of 200 µs was 500 Hz at the deposition-averaged target power density of 52 Wcm-2 (the peak target power density was 1100 Wcm-2). The Ar partial pressure was 2 Pa and the oxygen partial pressure oscillated between 0 and 0.08 Pa. Under these conditions, the deposition rate of ZrO2 films up to 120 nm/min was achieved on a floating substrate in the distance of 100 mm from the target.
From the time evolutions of the excited-state populations for the chosen atoms (Zr, Ar and O) and ions (Zr+, Zr2+, Ar+ and O+), and of the excitation temperature during a voltage pulse, the trends in a time evolution of the local ground-state densities of these atoms and ions during the voltage pulse were derived [3].
Near the target, a decrease in the ground-state densities of Ar and O atoms, caused by a gas rarefaction (due to a momentum and heat transfer during collisions with sputtered Zr atoms) and intense electron-impact ionization, was observed in the first half of the voltage pulse. Simultaneous, very effective electron-impact ionization of sputtered Zr atoms was proved. Moreover, it was found that a composition of particle fluxes onto the substrate during a film deposition is almost independent of the instantaneous oxygen partial pressure. Therefore, no multilayered structures of the films produced using this feed-back controlled technique, which could hypothetically result from the O2 flow pulsing, were observed.

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Abstract 26

CALORIMETRIC STUDY OF SECONDARY ELECTRONS IN SPUTTERING AND NITRIDING PIII PROCESSES

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Reactive deposition processes are commonly used in industry to guarantee high quality coatings with versatile properties. Modern technologies including magnetron sputtering and HIPIMS crucially depend on plasma properties including secondary electron emission from surfaces exposed to the plasma or energetic ion bombardment. The latter one can actually lead to changes in surface composition due to preferential sputtering or ion implantation. This effect is normally not accessible to conventional measurements of secondary electron coefficients
In this work, an approach using a passive calorimetric probe to investigate the effect of different stages of a substrate is presented using a plasma immersion ion implantation (PIII) setup where energetic ions are accelerated to the substrate. In preparatory work studies on the energy flux from the substrate during a PIII pulse were carried out which conclusively left only secondary electron emission as the only viable candidate for intense energy influx [1]. We measured several materials and alloys (e.g. Al, AlMg3, Mg, MgAl9Zn1, Cu, Zn, Mo, Ti and stainless steel 304) starting from a fully oxidized substrate using Ar sputtering into a clean metal state and afterwards nitriding towards a fully nitrided substrate. Thus, kinetics on the de-oxidizing and nitriding as well as relative secondary electron emission coefficients for the materials were obtained.

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Abstract 27

The Thermal Oxidation of TiAlN High Power Impulse Magnetron Sputtering Hard Coatings as Revealed by Combined Ion and Electron Spectroscopy

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The oxidation of Ti0.5Al0.5N hard coatings deposited by High Power Impulse Magnetron Sputtering were investigated at pressures ranging from 10-6 to 10-2 Pa and at temperatures of 298 and 800 K [1]. In vacuo X-ray Photoelectron Spectroscopy and Low Energy Ion Scattering revealed selective chemisorption of oxygen to Ti surface sites during the early stages of oxidation [2]. This process was further accompanied by the simultaneous formation of dissolved oxygen within the near surface region. Higher pressures and temperatures resulted into growth of a double layered surface oxide composed of a TiAl(O,N) growth region terminated by a Ti(IV) containing surface oxide [3,4]. As validated by Wagner plot analysis, the structure of the Ti(IV) surface oxide depends on the temperature of oxidation and was a mixed TiAlO after oxidation at 800 K and segregated (TiO2)(Al2O3) after oxidation at 298 K. Complementary Ultraviolet Photoelectron Spectroscopy revealed a high degree of nitrogen doping in both cases.

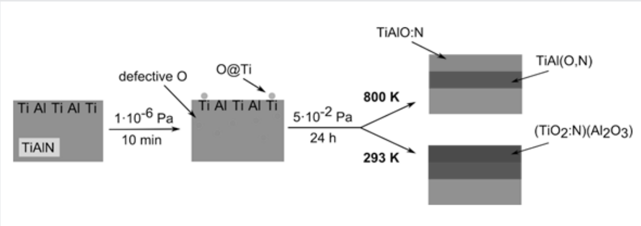

Figure 1: Surface oxide structure during different stages of the thermal oxidation of Ti0.5Al0.5N.

The results are of high relevance for the design of multi-layered nitridic hard coatings and for a fundamental understanding of the oxidation resistance of nitridic hard coatings.

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Abstract 28

RAMAN SPECTROSCOPY OF TITANIUM NITRIDE FILMS DEPOSITED BY REACTIVE MAGNETRON SPUTTERING WITH A HOT TARGET

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Films in the Ti-N system attract the most attention among nitrides. According to the Scopus data base around 3000 articles on this subject were published over the last five years. These films are promising for modifying the material surface properties in order to strengthen mechanical and corrosion resistance. In silicon technology they are used as ohmic and rectifying contacts, diffusion barriers for aluminum and copper metallization. TiN films are synthesized by variety of HiPIMS and, in particular, by reactive magnetron sputtering of a hot target, which could be heated to the melting point.
Using Raman spectroscopy, in this study we investigated how the N2 flow in the range of 2-6 sccm and the current density of discharge of magnetron with a hot target in the range of 10-40 mA/cm2 influence the TiN films chemical composition. Samples were prepared according to the three-level experiment plan on the cube of 32 -type, which was selected assuming that the impact of the process independent variables on the films' properties could be described in terms of polynomial model, which is not higher than the second order.
Fig. 1 shows the Raman spectra of three samples. They have wide absorption bands with maxima near 200, 310, which correspond to transverse and longitudinal acoustic and phonon modes. Modes at 440 и 550 cm-1

(Fig. 2) are attributed to vibration of the heavy atoms of titanium. Presence of the Raman scattering indicates the presence of point defects, which can be found even in the stoichiometric TiN samples. The band with maxima near 440 cm-1 corresponds to the acoustic oscillations of the second order, and with maxima near 550 cm-1 – to transverse optical phonon mode, which is associated with oscillations of the light ions of nitrogen.
The research findings are generally consistent with the known results for TiN films. The large width of the lines shows that films are polycrystalline. Detailed investigation of the Raman spectra allowed to establish the synthesis conditions for the most perfect films.

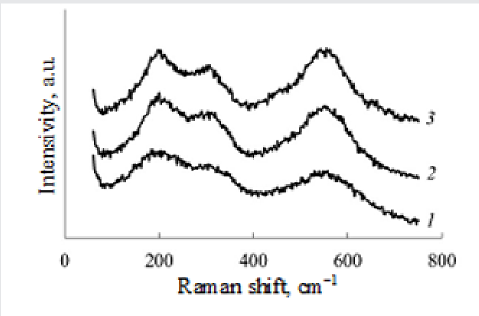

Fig. 1. Raman spectra of films TiN, deposited at current density 32 mA/cm2 and flow of N2 (sccm): 1 – 2; 2 – 4; 3 – 6;

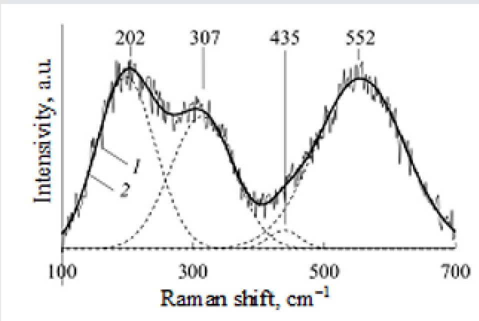

Fig. 2. Raman spectra of the sample 2 (fig. 1) and the result of the simulation: 1 – the measured spectrum; 2 – model

The study is supported by Russian Science Foundation (grant 15-19-00076).

Abstract 29

OXYNITRIDES FILMS MODEL SYNTHESIS BY THE HIGH POWER REACTIVE SPUTTERING TECHNIQUE

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[1] A.A. Barybin, V.I. Shapovalov, J. Appl. Phys. 101 (2007) 054905
[2] A.A. Barybin, A.V. Zav'alov, V.I. Shapovalov, Glass Phys. Chem., 38 (2012) 396

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For effective use of reactive sputtering it's needed to find its general regularities and relations between controlled and dependent variables. These investigations are conducted both experimentally and by simulation.
When high-power sputtering (HiPIMS) is used, the surface of the target heats up to the melting point. Heating could be achieved by high-power microsecond impulses or by direct current in a specifically designed magnetron with a hot target. When temperature increases target evaporation and thermionic emission influence the synthesis significantly.
Earlier we have developed physicochemical model of oxide1 and oxynitride2 films synthesis by dc reactive sputtering of a cold target. In this study, the model for sputtering of a hot target was developed.
In the model it is assumed that during the sputtering of a hot metal target in a Ar+O2+N2 environment, metal oxynitride appears as a solid solution (1–x)MmOn+xMpNs (0≤x≤1) on all surfaces inside the vacuum chamber. The components of the solution are formed by two independent surface chemical reactions. The main independent parameters of the process are the discharge current and the reactive gas flowrate. The thermionic emission from the target is taken into account in the discharge current. Partial pressures of reactive gases are taken as the main dependent variables. On the surface of the target there are

two competing processes: the formation of the thin layer of oxynitride and its removal by sputtering and evaporation. Substrate surfaces and chamber walls are passive. They change due to two chemical reactions and flow of the particles sputtering and evaporating from the target surface,

The full analytical description of the process of reactive sputtering is done using a system of sixteen algebraic equations, including equations of steady states of all surfaces, gas balance equations and equations that describe the gas flows on all surfaces.
Numerical solution of the system of equations allowed to obtain process describing dependencies that have a form of the s-shaped curves, containing areas with negative derivative.

Abstract 30

NON-CONTACT METHOD OF TEMPERATURE MEASURING OF TARGET SURFACE IN HIGH POWER MAGNETRON SPUTTERING

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High power magnetron sputtering, including impulse sputtering (HiPIMS), is characterized by heating of a target's surface up to high temperatures. The temperature of the target can have a significant impact on the processes occurring in the gas discharge and on surfaces of the target. Therefore, its measuring and control is of considerable practical interest. However, direct temperature measurement is a complicated technical task as well as the use of contactless pyrometric methods, which gives significant error.

To control the processes taking place during the sputtering of a target in a gas discharge plasma, traditionally used method of optical emission spectroscopy is utilized. As shown in Figure 1a, with a sufficient increase of the power, spectrum of the gas discharge undergoes significant changes. The recorded optical spectrum is a combination of the emission spectra of glow discharge and heat emission from the target. The method of estimating of temperature developed by authors is based on a comparison of the theoretical spectrum of blackbody radiation at a given temperature with the position of the minima on the entire spectrum (Figure 1b).

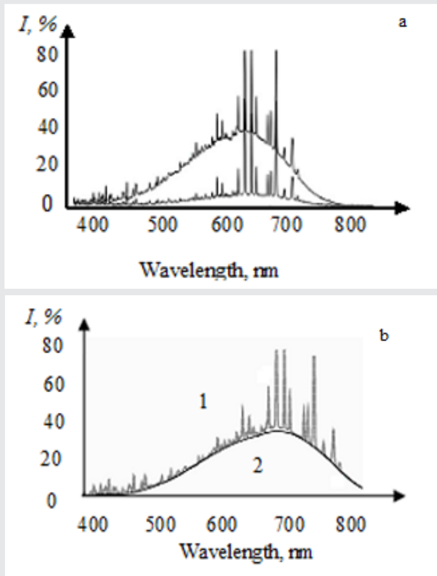

Fig.1. a – Typical emission spectra at different discharge power; b – Extraction of the thermal radiation spectrum (1 - initial spectrum, 2 – extracted spectra of thermal radiation).

Comparison of the experimentally determined values of temperature with the results of simulation of thermal processes in the COMSOL Multiphysics package showed that the difference does not exceed 10%.
Disadvantage of the method is that precise extraction of the spectrum of thermal radiation is not possible. Main reason for it are noises of the photodetector, which results in an error in determination of positions of minima. However, this disadvantage can be partially offset by smoothing the selected signal that will minimizes effect of single errors in determination of amplitude.

The study is supported by Russian Science Foundation (grant 15-19-00076).

Abstract 31

Crystalline deposition of GaN and ternary compounds by Pulsed Sputter Deposition of GaN

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GaN, being the material basis for modern LED lighting as well as high frequency HEMT transistor technology, is almost entirely based on metal organic chemical vapor deposition as suitable epitaxy technique. Recently, plasma techniques are discussed as a potential alternative, possibly allowing much lower growth temperatures and a much better scaling. In contrast to MOCVD related work, however, the development of plasma deposition as epitaxial technique for GaN is still in its infancy.
Here, we present results of epitaxially grown III-nitride layers and ternary compounds (AlGaN, InGaN) fabricated by pulsed sputter deposition.
The HiPIMS method is known to be able to substitute the thermal energy by the ion energies of the atoms that are needed for the crystalline growth. Therefore the process could be conducted at much lower temperatures, significantly enlarging the process window.
A setup to co-sputter ternary compounds as well as doped layers from pure metallic sources has been designed. 2 inch magnetron sources sputtering on a 4 inch substrates have been used in a sputter up geometry. The sources could be equipped with crucibles to hold liquid gallium as a target material. The quality of the GaN layers has been analyzed by different techniques, the correlation with growth parameters has been investigated. One result is that the quality of the material is strongly depending on the impurity level in the chamber. In the present configuration, a base pressure below 5*10-9 mbar can be reached. A clear correlation between thin film properties and the impurity levels in the vacuum chamber, which has been analyzed by quadrupol mass spectrometry, has been found.
Also the properties of a HiPIMS 4“ magnetron source in split geometry will be discussed, which has been designed for our process by CREAVAC. Due to the low melting point of Ga the HiPIMS process is quite critical and can e.g. result in droplet formation.

Abstract 32

HPMF process of Al-doped zinc oxide films from rotatable targets

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The constant price pressure on thin film CIGS solar modules causes a need for a technology which delivers a highly transparent and conductive oxide (TCO) at low cost. Nowadays the conventional sputtering of ZnO:Al2O3 (AZO) from ceramic targets leads the state of the art TCO. Reactive sputtering of metallic Zn:Al targets offers an attractive alternative for further price reduction. The reactive sputtering of ZnO:Al films with HIPIMS technology has shown a higher Damp Heat stability of solar cells, however unfortunately with low growth rates. The HIPIMS superimposed

midfrequency sputtering (HPMF) isa superimposed pulse pattern, which combines bipolar HIPIMS and a bipolar mid-frequency (MF) process on the same double cathode. This process has the chance to keep the high Damp Heat at high growth rates. In the present work an industrial relevant rotatable magnetron set-up in an in-line coater was built to show the possibility of up-scaling and thoroughly controlling the reactive HPMF process. The process conditions as well as the resulting AZO films will be discussed.

Abstract 33

The effect of annealing on mechanical properties and constitution of TiC:H and TiC/a-C:H thin films deposited by high power impulse magnetron sputtering

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Single-phase nanocrystalline hydrogen–containing titanium carbide thin films and nanocomposites consisting of nanocrystalline titanium carbide and amorphous hydrogenated carbon phases were deposited by reactive high power impulse magnetron sputtering, using a titanium target and a gas mixture of argon and methane. The constitution, microstructure and selected mechanical properties of as-deposited films grown at different reactive gas flows without external substrate-heating were thoroughly investigated. A rise of the methane gas flow at a constant argon gas flow leads to an increase of the carbon content in the thin film up to 63 at.%. The microstructure develops from nanocrystalline titanium, over titanium carbide to a nanocomposite. The hardness shows a maximum of about 3000 HV0.05 for a nearly stoichiometric titanium carbide composition. A thin film with a carbon to titanium ratio of close to 1 and the thin film with the highest carbon content were chosen for further investigations in terms of their temperature stability. Annealing was performed consecutively stepwise from 100°C to 1000°C at 10-4 Pa for 2 h. Analyses of the constitution, microstructure and mechanical properties include electron probe micro analysis, Raman spectroscopy, X-ray diffraction in Bragg-Brentano-geometry, Vickers micro-hardness, and stress measurements based on Stoney`s formula. At elevated temperatures of 800°C the titanium carbide grains grow in size as seen by X-ray diffraction line broadening for both the samples with a carbon to titanium ratio of close to 1 as well as the carbon-based nanocomposite. Raman spectroscopy of the carbon-based nanocomposite also reveals an increasing graphitization. This structural changes correlate well with a decrease in the Vickers micro-hardness.

Abstract 34

Measuring the Ionized Fraction of Film Forming species

Gerdes, H.*[1]; Spreemann, D.[1]; Bandorf, R.[1] ; Vergöhl, M.[1]; Bräuer, G.[1]

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In sputter processes the resulting film properties are depending on many process parameters like working pressure, target substrate distance, gas flows etc. Especially, when using High Power Impuls Magnetron Sputtering the fraction of ions arriving at the substrate will influence the growths of the film.
This presentation will focus on measuring the ionized fraction of the film forming species. Therefore, a commercial gridded Quartz Crystal Microbalance (gQCM) was used in a sputtering process of cooper and aluminum and

was placed at the substrate level. The results were also compared to optical emission spectroscopy and Langmuir measurements. The investigations showed that with low peak currents mainly the neutrals determine the film growth, while for higher peak currents the ionized materials made nearly 50 percent of the film growth.

Abstract 35

Metal-doped DLC layers prepared by HIPIMS/PECVD

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DLC (diamond-like-carbon) is widely used in technical applications for its wear resistance but also for its sensory properties. Depending on the process parameters, the properties of DLC can be varied in a wide range, from graphitic phases to more diamond like phases. In addition, the electrical properties can be influenced by doping. For example doping with metals like nickel is known to increase the electrical conductivity, decrease the thermal coefficient of electrical resistance (TCR), and enhance the strain sensitivity, i.e. the gauge factor of DLC layers. Metal doped DLC films, using biocompatible metals (Nb, Zr) are investigated. The films are prepared in a reactive HIPIMS process using acetylene as hydrocarbon-precursor. The influence of the preparation condition on morphology (SEM), metal content, as well as on the electric behavior, i.e. electrical conductivity, TCR, and gauge factor will be studied.

Abstract 36

Investigation of the ion to neutral ratio by plasma emission monitoring using metallic and reactive HIPIMS process

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In recent years, the focus of research in High Power Impulse Magnetron Sputtering (HIPIMS) has shifted to reactive processes. In contrast to the often-referred reduction in deposition rate by metallic HIPIMS, there are several publications indicating that for reactive HIPIMS similar or even higher deposition rates for films with superior properties are possible. Increasing the peak current in the discharge will lead to formation of ions at different ionization stages. Reactive optical process control usually monitors the reduction of the intensity of a characteristic metal emission line. While this is fine and well established in conventional DC and MF sputtering, the question arises which effects contribute to the reduction of the emission intensity and how valid the information is for defined process control in HIPIMS. Besides the reduction by the added reactive gas, the intensity also decreases by ionizing some of the metal species. Using titanium as well studied model material the influence of ionization on the change of the emission intensity of characteristic lines will be discussed. First, the influence will be studied for pure metallic processes. Secondly, the influence of additional reactive gas, in this case of oxygen, will be investigated and discussed correspondingly. Finally, the idea is to point out options to control processes for a given stoichiometry and to adjust independently the amount of film forming metal ions in the process.

ABSTRACT 37

Coatings for Friction Stir Welding Applications

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ABSTRACT 38

Influence of ion-to-metal flux ratio on the mechanical and tribological properties of TiN coatings deposited by HiPIMS

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High power impulse magnetron sputtering (HiPIMS), due to its dense plasma and high ionization degree of sputtered material, enables to deposit high-quality thin films, with fully dense structure, low RMS surface roughness and superior hardness and wear properties. Operating the HiPIMS discharge with ultra-short pulses opens the possibility to control the degree of metal ionization and to tailor the microstructure of the growing film.

In order to study the influence of ion-to-metal flux ratio on the thin film's microstructure, nanocrystalline titanium nitride (TiN) thin films were deposited on silicon substrates at different degrees of metal ionization by adjusting the HIPIMS pulse duration and operation mode (single or multi-pulse). Energy resolved mass spectrometry and quartz crystal microbalance in combination with a two-gridded energy analyzer have been used to determine the ion energy distributions, composition and fraction of ionized metal species in the substrate's vicinity. The properties of deposition flux were correlated to the topological, structural, mechanical and tribological properties (hardness, Young's modulus, adhesion/cohesion, coefficient of friction) of TiN thin films. Atomic force microscopy, scanning electron microscopy, nanoindentation and nanoscratch measurements were carried out in order to study the surface topography, microstructure, mechanical and tribological properties of TiN thin films. Structural properties were analyzed by X-ray diffraction.

According to the mass spectrometryy results, the contribution of low and intermediate energetic parts (ions with energies below 20 eV) to Ti+ ion energy distribution functions significantly increases during ultra-short and multi-pulse HiPIMS discharge. The highest Ti ion flux fraction (up to 50%) was measured for the multi-pulse HiPIMS operation modes.

The table below presents values of hardness (H), absolute Young's modulus (E), coefficient of friction (μ – nanoscratch, COF – ball-on-disk test) and critical normal forces (LC1 and LC2) corresponding to 500 nm nanocrystalline TiN thin films deposited by short-HiPIMS (4-16 μs) and m-HiPIMS (3x4 μs) on not intentionally heated silicon substrates, at room temperature.

The hardest coatings with the highest H/E ratio were found to be the TiN thin films deposited by m-HiPIMS. These samples are also characterized by the lowest coefficient of friction.

Sample	TiN_1	TiN_2	TiN_3	TiN_4	TiN_5
pw (us)	4	8	12	16	3x4
H (GPa)	15.1 ± 1.1	16.1 ± 0.9	18.6 ± 1.3	17.2 ± 1.1	22.3 ± 1.1
E (GPa)	338.1 ± 22.5	344.8 ± 19.6	363.1 ± 9.7	384.1 ± 31.4	351.6 ± 35.9
H / E	0.045	0.047	0.051	0.045	0.063
µ	0.06	0.06	0.6	0.05	0.05
COF	0.42	0.50	0.33	0.53	0.26
F - LC_1 (N)	3.8	4.4	3.9	4.5	4.1
F - LC_2 (N)	8.2	6.2	7.4	6.1	5.6

Table 1

Abstract 39

Velocity distribution of sputtered species in the ionization region

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Mass spectrometers and Langmuir probes are often used to measure energy distribution functions and densities of charged species in high power impulse magnetron sputtering (HiPIMS) discharges. However, these tools disturb the plasma and are therefore not suitable for measurements in the ionization region close to the target, where the magnetic field lines are closed. In this work, optical emission spectroscopy was used as a non-invasive alternative. A high resolution plane grating spectrograph combined with a fast, gated, intensified CCD camera was used to investigate the discharge. The shape of the optical emission lines of sputtered species was studied. Doppler broadening and shift was analyzed to gain information about the velocity distribution of sputtered species in the ionization region of the discharge. As a result, evidence of strong self-scattering in the ionization region was found. For the studied parameter range, the velocity distribution was found to be independent on the applied voltage and constant during the discharge pulse.

Abstract 40

High mobility amorphous zinc oxynitride films depos-ited by reactive-HiPIMS

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Zinc oxynitride films, has been considered as one of the strong substitute to conventional semicon-ductor films like Si and InGaZnO due to its high electron Hall mobility. However, owing to the low binding energy of zinc with nitrogen, zinc oxynitride films deposited by direct current magnetron sputtering suffer from the instability in their chemical composition, which subsequently affects the functionality of films in the semiconductor devices. Although post-deposition processing such as ar-gon plasma treatment has been implemented to improve the film performance, the formation of nanocrystallites in the postdeposition process would create procleation paths in the films and consequently detriments the device performance. Hence depositing zinc oxide films with high-mobility and high-stability is always a challenge. In this work, using reactive HiPIMS, we demonstrate the deposi-tion of amorphous zinc oxynitride films exhibiting high electron Hall mobility greater than 172 cm2V-1s-1 based on the detailed investigation of the plasma discharge and chemical condition of the target surface. After ensuring good process stability and negating any feedback control in Ar/O2/N2 reactive high power impulse magnetron sputtering (HiPIMS) of zinc target, suitable pulse parameters have been chosen to deposit amorphous zinc oxynitride films with high nitrogen concentration and high electron Hall mobility. The increase in nitrogen concentration in the films is more dependent of the pulse length than the nitrogen partial pressure in the gas mixture. We also show that the change in chemical condition of target alters the energy and flux of deposits impacting on film surface, and in-directly

influences the film density and surface roughness of the zinc oxynitride films deposited at dif-ferent angles with respect to the target. High-mobility and high-stability in zinc oxynitride films can be achieved by depositing denser films without nanocrystallties, which is possible by tuning the pulse characteristics and angular position of substrate with respect to the target in Ar/O2/N2 reactive-HiPIMS of zinc target.

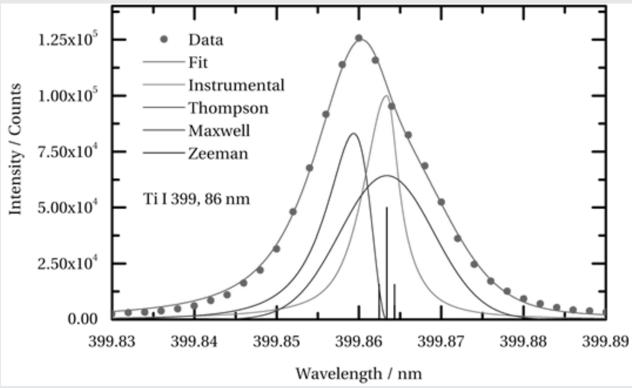

Fig. 1: Measured and fitted Titanium neutral emission line with individual components of the fit function

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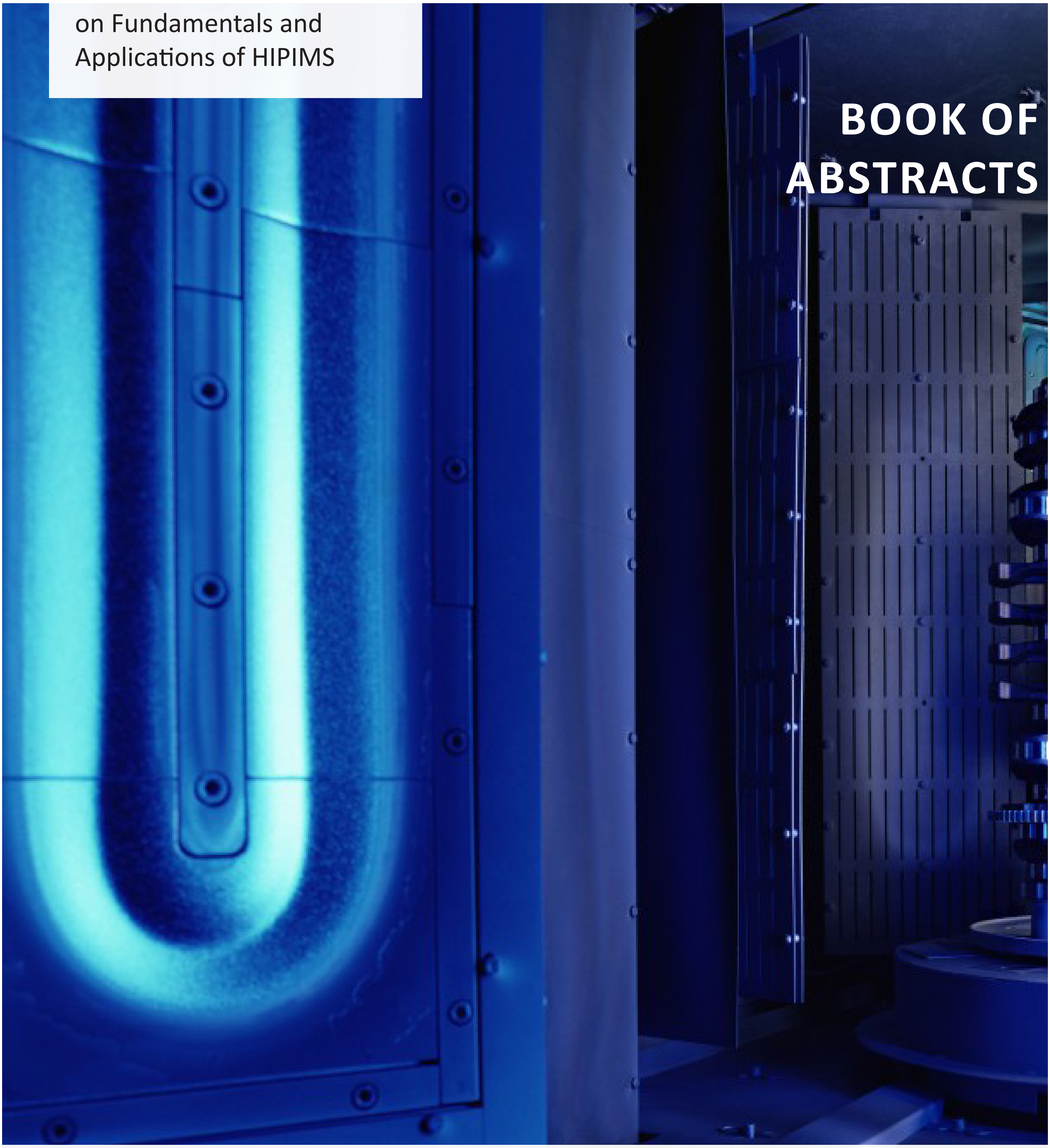

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