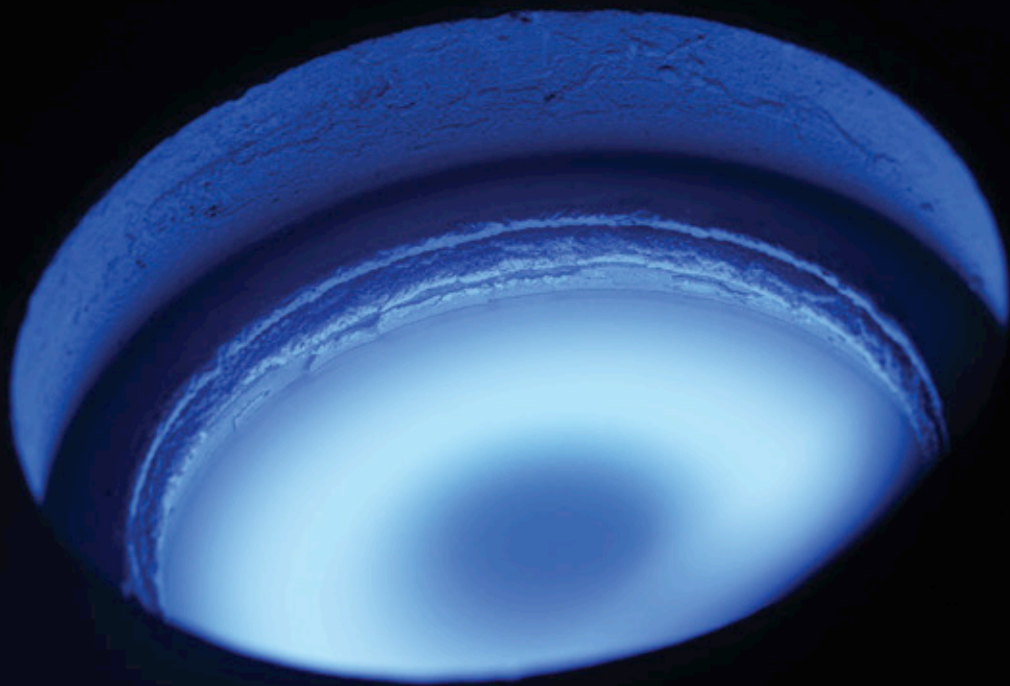


Ninth International Conference on HIPIMS *Abstract Book*



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HIPIMS superimposed sputtering 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) is a 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.

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Low-temperature (300°C) HiPIMS deposition of thermochromic VO₂ films with antireflection SiO₂ overlayers

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Vanadium dioxide (VO₂) is a technologically important thin film material of a high current world wide interest due to its reversible first-order thermochromic transition relatively near room temperature (approximately 68°C).

In this work, reactive HiPIMS with a pulsed O₂ flow control and to-substrate O₂ injection into a high-density plasma in front of the sputtered vanadium target was used for low-temperature (300°C) deposition of VO₂ films with a pronounced semiconductor-to-metal transition onto conventional soda-lime glass substrates without any substrate bias voltage and without any interlayer. The depositions were performed using an unbalanced magnetron with a planar target of 50.8 mm diameter in argon-oxygen gas mixtures at the argon pressure of 1 Pa. The deposition-averaged target power density was close to 13 Wcm⁻² at a duty cycle of 1% with a peak target power density up to 5 kWcm⁻² during 50 μs voltage pulses. A high modulation of the transmittance at 2500 nm (between 51% and 8% at the film thickness of 88 nm) and the electrical resistivity (changed 350 times) at the transition temperature of 56-57°C was achieved for the VO₂ films synthesized at the controlled oscillations of the oxygen partial pressure around 15 mPa. Under these conditions, appropriate composition of the total ion flux and higher ion energies (up to 50 eV relative to ground potential) supported crystallization of the thermochromic phase (VO₂(R) during the deposition and VO₂(M1) at the room temperature).

Antireflection SiO₂ layers were deposited using mid-frequency (50 kHz, duty cycle of 50% and deposition-averaged target power density close to 8 Wcm⁻²) bipolar dual magnetron sputtering onto the top of VO₂ layers at a surface temperature below 35°C in order to improve the optical and mechanical performance. We focus on the dependence of the luminous transmittance, T_{lum} , and the modulation of the solar transmittance, ΔT_{sol} , on the SiO₂ layer thickness. We show an improvement due to the SiO₂ overlayer of up to 16% (from 40.3% to 56.3%) for T_{lum} and up to 2.6% (from 7.7% to 10.3%) for ΔT_{sol} .

The results are important for the design and low-temperature fabrication of high-performance durable thermochromic VO₂-based coatings for smart window applications.

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Codeposition of Silicon-doped tantalum oxide films by HiPIMS for tunable resistive switching applications

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Thin film Si-doped Tantalum oxide has been energetically deposited at 270 °C using high power impulse magnetron sputtering (HiPIMS). Reactive co-deposition from Ta (HiPIMS mode) and Si (DC magnetron sputtering mode) targets yielded a gradient in the Ta:Si ratio across a 4-inch substrate ranging the Si incorporation from 2 to 9.5%. The films were deposited on SiO₂/Si substrate with platinum electrodes patterned on it. The switching characteristics, film density and optical properties of the films were studied as a function of composition. The optimum value of Si doping in tantalum oxide was found to be about 5.2 %. The cross bar memory devices made from such composition showed larger range of dynamic conductance with access to the intermediate conducting states and also exhibited high cycling endurance (>10¹² set and reset). The doped Si atoms could possibly facilitate the formation of oxygen vacancies and their mobility in the switching layer with self-regulated ion hopping distance and drift velocity. On contrary, the resistive switching functionalities were compromised for the devices made from the reactive co-deposition from Si (HiPIMS mode) and Ta (DC magnetron sputtering mode) targets with a similar Ta:Si composition. The RBS and electrical switching characterization results suggest that HiPIMS can produce dense, high quality Si-doped Ta₂O_{5-x} films suitable for resistive switching applications.

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Reactive deep oscillation magnetron sputtering of Al-O-N films with tunable composition and properties

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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 [1,2].

In this work, a modified version of HiPIMS, called Deep Oscillation Magnetron Sputtering, 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 Al-O-N films with a tunable elemental composition, structure and properties. We give the basic principles of this method, maximizing the degree of dissociation of both O₂ and N₂ molecules in a discharge plasma, which leads to a replacement of very different reactivities of the O₂ and N₂ molecules with metal atoms on the surface of growing films by similar (high) reactivities of atomic O and N.

The depositions were performed using a strongly unbalanced magnetron with a planar aluminium 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 %. Voltage macropulses, composed of 10 voltage micropulses (pulse-on time of 20 μs and pulse-off time of 30 μs), with a total length of 500 μs and repetition frequency of 350 Hz were used for all depositions with a maximum target power density up to 675 Wcm⁻² during pulses at a deposition-averaged target power density of 8.5 Wcm⁻². The substrate temperatures were less than 120°C (no external heater) during the depositions of films on a floating substrate at the distance of 100 mm from the target. A pulsed reactive gas (O₂ and N₂) flow control made it possible to produce hard (13–19 GPa) and highly optically transparent (extinction coefficient ≤ 1 × 10⁻⁴ at 550 nm) Al-O-N films with gradually changed elemental compositions from Al₂O₃ into AlN.

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Current peak profiles and plasma optical emission spectra during HiPIMS of Zn/Al in an Ar and Ar/O₂ atmosphere

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Transparent conducting oxide (TCO) thin films are essential and widely used material in optoelectronics. Among the TCOs aluminium doped zinc oxide (AZO) is one of the most studied and used material due to the promising optical and electrical properties. Although HiPIMS technology is a well-established thin film deposition technique, there are not a lot of scientific papers about TCO films deposition by the HiPIMS. In the HiPIMS regime ionised sputtered atoms could be used to control delivered energy to the growing film by a negative and synchronized bias voltage which could also prevent the unfavorable contribution from negative oxygen ions in reactive sputtering. The reactive sputtering is often used to deposit compound thin films; however, a precise process control is required because the desirable properties together with a sufficient deposition rate usually could only be obtained in the narrow reactive gas partial pressure range. The change in the current peak profile with the reactive gas could be used as a control parameter.

In this study the Zn/Al (98:2 wt.%) target was sputtered by rare high power pulses (power unit Melec SIPP2000) in an Ar and Ar + O₂ atmosphere. The profiles of peak current and the optical emission spectra collected 2 cm above the target surface were detected as a function of electrical parameters (frequency, pulse time, average power) and oxygen flow.

Plasma emission spectrum changes from mainly excited Ar emission to excited Zn emission when the frequency is decreased. That could indicate the rarefaction of argon gas. The plasma impedance decreases and the peak current increases sharply when the peak power of 0.2 kW/cm² is reached. At this value the ionization of sputtered Zn atoms starts. The current value in the end of the pulse (500 μs) starts to decrease but in the middle increase when the oxygen flow is increased.

The results obtained in this study could be used to better understand and optimized the growth condition of AZO films by reactive HiPIMS. Additionally, Zn sputtering in the HiPIMS regime is not widely studied as well.

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Erosion performance of CrAlYN/CrN nanoscale multilayer coatings deposited on Ti6Al4V by HIPIMS

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Water droplet erosion (WDE) protection of Ti6Al4V turbofan blades has recently attracted the attention of the aviation industry. In this work the performance of a novel CrAlYN/CrN coating utilising nanoscale multilayer structure was evaluated as a potential candidate for this demanding application.

The coating was deposited on mechanically polished coupons from Ti6Al4V alloy by High Power Impulse Magnetron Sputtering (HIPIMS) technology. The coatings were produced on a HIPIMS enabled Hauzer HTC1000-4 coater using dedicated magnetron and substrate bias power supplies by Huettinger.

Low angle XRD and TEM analyses revealed coatings nanoscale multilayer structure with by-layer thickness of $\Delta = 2.75$ nm. Glancing angle XRD and far angle $\theta - 2\theta$ scans showed that the coatings crystallised into a B1 NaCl f.c.c. structure and exhibited {111} preferred crystallographic texture.

Scratch adhesion tests using a REVETEST instrument showed enhanced adhesion, with critical load value of $L_c > 80$ N due to the application of HIPIMS surface pre-treatment.

Previous research indicates a correlation in material performance ranking under the WDE and cavitation erosion (CE) tests. The latter has been proposed as a screening process in literature and ASTM G73. Hence, the WDE performance of a novel nanoscale multilayer structured CrAlYN/CrN coating on Ti6Al4V was investigated by means of CE. The tests were conducted with an ultrasonic cavitation device (UIP 1000 hdt) in distilled water at room temperature ($\sim 25 \pm 2$ °C). Cavitation was induced by longitudinal oscillation of a vibrating tip of a titanium sonotrode at 20 kHz at a peak-to-peak amplitude of 50 μ m.

Results of the CE tests showed a significant improvement in the resistance of the coated specimens compared to the bare Ti6Al4V substrate. The erosion rate of the coated specimens was limited to 0.004 mg/min after 270 mins of CE test, compared to 0.260 mg/min for the substrate material. The most common failure mode is through detachment and is exhibited by other coating systems under the same test conditions. In contrast multilayer coatings demonstrated excellent bonding strength and resistance when subjected to CE. Focused ion beam (FIB) investigations on cross-sections taken from samples subjected to a full CA test showed clearly that cracks are deflected at the interfaces of the individual layers in the nanoscale multilayer stack, which mechanism significantly retards the overall coating damage. However, it was revealed that nodular, open void and corn-shape defects which are formed during the deposition process on coating surfaces can potentially act as shear stress concentrators and enhance erosion rates locally. SEM and FIB cross sectional analyses further revealed crack formation in the Ti6Al4V alloy in the region immediately beneath the coating substrate interface. The origin of such cracks is believed to be due to the exhaustion of the fatigue limit of the base material.

The paper discusses the possible damage mechanisms observed in nanoscale multilayer coatings when subjected to cavitation erosion attack.

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Spoke formation in large scale rectangular magnetrons

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Rectangular magnetron cathodes are widely used in the sputter deposition industry, however spokes have been studied mainly in circular target geometry. The variation in magnetic field strength between the corners and straight sections of the cathode as well as the smaller spoke-to-cathode size ratio influence the motion and formation of the spokes. Fast camera imaging was used to study High Power Impulse Magnetron Sputtering (HIPIMS) discharges on 200×600 mm cathodes in a Hauzer HTC 1000/4 system. Spokes were observed at peak current densities as low as 0.6 Acm^{-2} . Spoke number (mode) was found to decrease with magnetic field whereas the velocity increased. Spokes were triangular for strong fields and diffusive for weaker fields for the same peak current of 1 Acm^{-2} . The splitting of spokes due to acceleration of a portion was observed. At low pressure the spoke shape was a diffuse triangle which widened and advanced in the $\mathbf{E} \times \mathbf{B}$ direction. The spokes turned diffusive at the corners and narrow sections of the magnetron and reformed upon re-entry into the straight sections. The shape of the spokes is generally triangular due to the sequential processes of build-up of ionisation to a critical value, rupture of the field and restoration of confinement. In strong confinement fields these processes are faster and produce triangular spokes. In weaker fields the triangle is stretched out resulting in larger volumes of escaping plasma. The behaviour of spokes in the corners is discussed in terms of electron dynamics and the weaker magnetic fields. At low pressures the spokes may be dominated by metal sputtering and ionisation which are initiated in the centre of the racetrack and spread across.

It is argued that spoke formation could be linked to the ratio of plasma density and magnetic field (beta, β). Spokes are associated with localised rupture in confinement and ejection of intense particle beams. Thus spokes are a route for the escape of plasma from the confinement field which leads to a greater degree of freedom, greater number of accessible states and greater entropy. It is suggested that the increase in entropy is the driving force for the creation of zones of intense ionisation which leads to high plasma pressure and localised rupture of the confinement field.

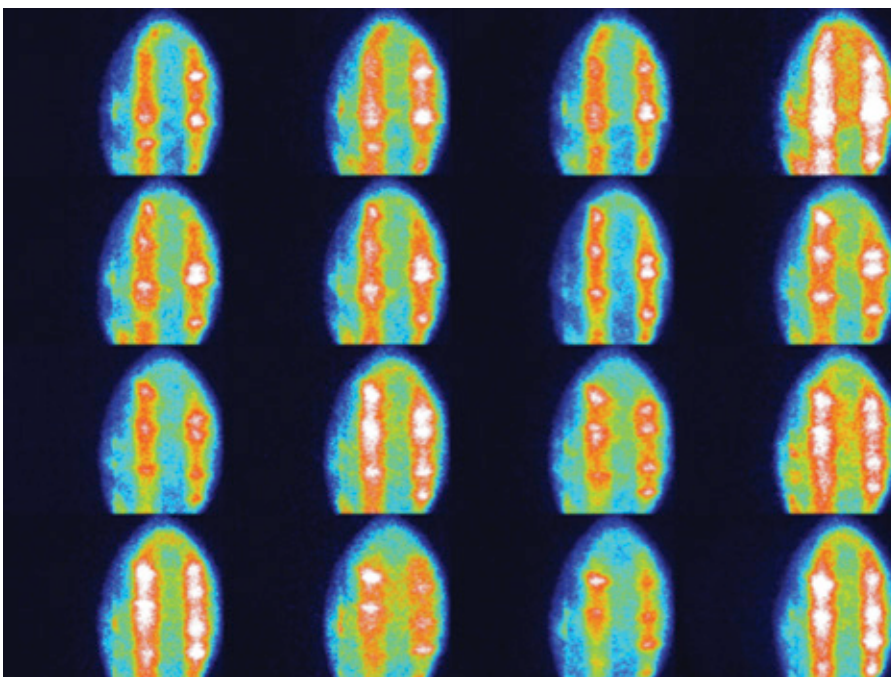


Fig. 1:

Fast camera, full-spectrum images of a 200×600 mm magnetron cathode in HIPIMS mode at peak current of 1500 A, peak voltage of -1000 V and Ar pressure of 0.35 Pa.

Exposure is 200 ns and inter-frame period is 1000 ns.

Observed are spoke splitting and spoke restoration upon re-entry into the magnetron's straight section.

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Structural and mechanical properties of DLC films deposited by Ne HiPIMS

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High Power Impulse Magnetron Sputtering (HiPIMS) has been under consideration for hard DLC thin films deposition in recent years [1-3]. In HiPIMS, a large fraction of sputtered atoms is ionized, thanks to 2–3 orders of magnitude higher plasma densities than in DCMS [4, 5]. The major driver to use HiPIMS for DLC deposition is the possibility of C ions formation in the plasma and subsequent use of these ions to bombard the substrate in a similar way to what is presently done in ARC deposition. The main strategy to achieve ionization of the sputtered species in HiPIMS is to promote electron impact ionization through increasing the plasma density. This route has been successfully implemented for many metals, i. e., for elements which exhibit ionization energies between 6 and 8 eV. However, this strategy is not effective for C which exhibit a significantly higher ionization energy (11.6 eV) and lower ionization cross-section. As a result, in HiPIMS discharges with a C target the C⁺/C ratio does not exceed 5 % [6]. An alternative strategy to increase the ionization reaction of the sputtered carbon species, proposed by Aijaz et al., consists in increasing the electron temperature of the discharge [6]. This can be achieved by using gases with higher ionization energy than Ar (15.6 eV) such as Ne (21.56 eV) in the plasma.

Several authors have reported that adding Ne to the plasma is beneficial for the mechanical and tribological properties of DLC films [7, 8]. In this work the effect of adding Ne to a pure HiPIMS Ar discharge is investigated using a flat probe and correlated to the structural and mechanical properties of deposited DLC films.

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Graphene Growth by Using HIPIMS

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Tremendous interest has been attracted to graphene growth, due to its unique electronic, optical and mechanical properties. To date, many preparation routes for graphene layer growth have been demonstrated. However, most of the routes relied on the thermally assisted processes, either in-situ or post heat treatment, which require high temperature and result in limited practical usage. High power impulse magnetron sputtering (HIPIMS), known for generating high density plasma, is expected to energize carbon ions emitted from graphite cathode, and possibly produce graphene layer at low substrate temperature. In this study, HIPIMS was used to deposit graphene layers on copper foil. Substrate temperature was controlled to deposit graphene layers. Optical emission spectroscopy (OES) were used to exam HIPIMS plasma. The samples were characterized by using Raman spectroscopy and transmission electron microscopy (TEM).

Based on the experimental result, a substrate temperature below 600 °C would be able to produce graphene layers by using HIPIMS as opposed to DCMS. A 7-10 layer graphene on copper foil is illustrated by TEM high-resolution images, with its corresponding Raman spectrum showing 2D peak.

Keywords: Graphene, High power impulse magnetron sputtering, Copper substrate

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High sp^3 DLC with iPVD through Modified High Impulse Magnetron Sputtering

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High Power Impulse Magnetron Sputtering (HiPIMS) produces film qualities superior to that of Direct Current Magnetron Sputtering (DCMS) at the cost of deposition rate attributed to returned ions to the target surface. Ion fraction can be increased along with deposition rate by using a modified magnetic field structure with field strength dropping off by 90% within 5cm of the target surface. With controlled electron loss from the high magnetic field trap, a low density plasma is formed between the highly confined region and the substrate surface. The low density plasma ionizes sputtered target material and background gas increasing the total ion to neutral at the target surface. To utilize the increased ion fractions further from the target surface, a lower potential positive polarity pulse following the negative polarity high power pulse while operating at peak current densities in the HiPIMS regime is introduced. This further accelerates ion from the target surface that would otherwise be trapped in the magnetic field and controls the ion energy and anisotropy independent of the substrate condition.

Previous works has shown deposition rates increase by 5nm/s +/- 0.2nm/s for both DCMS and HiPIMS when utilizing the tight magnetic field configuration measured, while producing increases from 13% +/- 2% to 35% +/- 3% in total ion-neutral fraction 10cm from the target surface using copper. Introducing a positive kick resulted in improved film quality without manipulating the substrate condition. Residual tensile stress of copper on silicon reduced from 1000MPa +/- 150MPa to 350MPa +/- 50Mpa. Utilizing a modified magnetic field topology and/or the positive polarity kick pulse with a carbon discharge, DLC coatings can be tuned to grow high sp^3 fraction films without the introduction of hydrogen seen from growth using chemical growth methods. Increases in ion flux and energy from the positive kick pulse introduces reorganization during film growth. This work presents effects on plasma discharge of the positive kick pulse including plasma density, ion-neutral fraction, and total flux near the substrate surface. It then investigates the effects the increase in the plasma parameters have on film stress and crystallographic structure in a carbon film.

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Controlling stoichiometry and ionization of reactive HIPIMS processes by using plasma emission monitoring

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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. Despite a reduced hysteresis effect in the case of reactive HIPIMS, this process still has to be controlled regarding stoichiometry and ionization for achieving high film qualities, especially on larger scale.

While monitoring the reduction of the intensity of a characteristic metal emission line is well established in conventional DC and MF sputtering, the question arises which effects contribute to the reduction of the emission intensity and how valid these information's are for a defined process control in HIPIMS.

Using plasma monitoring and titanium as a well studied model material the correlation of optical emission and the measured intensity of neutral and ionized species was studied for reactive HIPIMS processes. Based on these findings an active optical control is shown, that allows for adjusting the working point, i.e. reactive gas flow and the ionization independently. For example, high values for transparency and refractive index are indicators of a correct stoichiometry ratio and good crystallinity due to a high degree of ionization. Additionally the resulting film properties will be discussed for different working points.

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HiPIMS meets diamond

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This paper will introduce a visionary new class of coating materials with revolutionary properties. It creates added value by merging diamond – the hardest of all materials – with HiPIMS – smooth and dense sputtered films – into one new material.

The starting point of HiPIMS meets Diamond was the search for new coatings for high gloss mirror finish machining with micro-cuttings tools for the 3C industry (Computers, Consumer Electronics & Communications). The extreme requirements set by this industry brought up a new approach of tuning the “knobs” of HiPIMS for novel plasma regimes during etching and the deposition phase. This involves new hardware features as individual parameter sets for all HiPIMS sources while keeping the synchronization to the dedicated HiPIMS bias. Application cases will show that the plasma synchronization is the key to minimize intrinsic stress while having a deposition rate as high as 2µm/h at the same time.

This new way of controlling the pulsed HiPIMS plasma made possible the design of HiPIMS meets Diamond. The exceptionally hard diamond provides the perfect foundation to the HiPIMS film. Furthermore, diamond has an outstanding thermal conductivity and spreads the extreme heat coming from the cutting zone. The smooth, droplet-free surface of HiPIMS coatings reduces friction, protects the diamond against oxidation and optimizes the running in process of the cutting tool. All this makes HiPIMS meets Diamond the perfect candidate for coatings for heatresistant superalloys.

Case studies as the machining of casted CrCo for medical implants and the milling of stacks with extra thick titanium layers show that the radically new materials concept HiPIMS meets Diamond paves the way for new business for cutting tools after the combustion engine.

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The implementation of a reversed voltage pulse technology enables enhanced coating properties for glass, plastics, fiber and other non-conductive substrates.

A special consideration of this work will be given to medical coatings of biocompatible polymers on implantable devices.

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HiPIMS (Hi-Power-Impulse-Magnetron-Sputtering), the state of the art deposition technology with its high degree of ionization, has shown its significant film property improvements (hardness, density, smoothness, refractory index, etc..) in many fields and applications. These advances are specifically possible for conductive substrates, where a bias voltage can be applied to attract the ions. Evidently, for glass, plastics or other non-conductive substrate materials, bias voltage is not effective.

Asymmetric bipolar pulsed DC, resp. a short voltage reversal pulse, has been introduced to state of the art reactive magnetron sputtering in the 90's. This technique has been intensively investigated and is well integrated into industrial high end coatings. Studies have shown that besides preventing target poisoning and dielectric arcing, there are a lot more advantages evident when an optimized positive voltage pulse is applied. Yet, if this positive pulse is supplemented to the HiPIMS technology with its high degree of ionized film forming materials, even greater add-on values to the deposition processes can be created. Definitely, the enhanced high energetic positive ion bombardment exploits its advantages also on nonconductive substrates where no bias voltage can be applied. Large area glass as well as temperature sensitive plastics or textiles can be processed with remarkable HiPIMS enhanced film properties at respectable deposition rates. Additionally, due to the raise of the plasma potential, higher incorporation of reactive species into the depositing film, optical property improvements and elastic hardness values can be observed.

Significant benefits for coating of biocompatible polymers, like polyimide or silicone, are expected. Especially for implantable polymer substrates, a good layer adhesion is strictly necessary to prevent delamination and system failures. Another big point is the immune response of the detached coating particles. HiPIMS technology with positive bipolar pulses can prevent those coating failures and improve the stability of active implantable medical devices.

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Molybdenum back contacts for photovoltaic applications deposited by HIPIMS

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The back contact of photovoltaic devices has a major influence on the efficiency of the cells. Back contacts need to have low resistivity to reduce energy losses and need to be as reflective as possible to allow for more photons to be absorbed. Molybdenum has been identified as a good candidate that fulfils both these requirements.

The influence of HIPIMS on the coating properties of Molybdenum on soda lime glass is not known.

In this study the effect of HIPIMS deposition parameters on the microstructure, texture, resistivity and reflectivity are examined and plasma parameters will be correlated with the coating properties.

A voltage-pulse time matrix was devised varying the voltage from 800 - 1500 V and the pulse time was increased from 60 - 1000 μ s in 5 steps. Processes were operated at 0.22 Pa (LP) and 0.44 Pa (HP).

The Mo II / Mo I ratio shows that the intensity is constant over all pulse time settings between 1150 V and 1350 V at LP, while for HP the ratio is constant over all voltages and decreases rapidly between 60-125 μ s. Mass spectroscopy measurements taken in the middle of the pulse; show a broad Mo²⁺ peak between 1 - 10 eV, which with increasing pulse time has a tail with an energy of up to 25 eV (Fig.1).

Single layer deposition at LP resulted in poor adhesion and low resistivity. At HP the adhesion was good with high resistivity. Multilayer coatings with a HP base layer and LP top layer were found to have good adhesion with lower resistivity than single HP layers.

SEM micrographs revealed that for increasing voltage the size of the rice like grain structure reduces with increasing voltage (Fig.2).

These results show that HIPIMS can be used effectively to deposit tailored Mo back contacts with good adhesion and low resistivity.

Keywords: HIPIMS, CIGS, Back contact

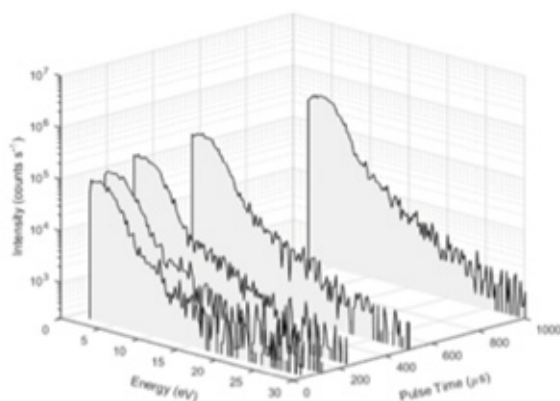


Fig. 1: Energy resolved MS of Mo²⁺ at 0.44 Pa and 1500 V.

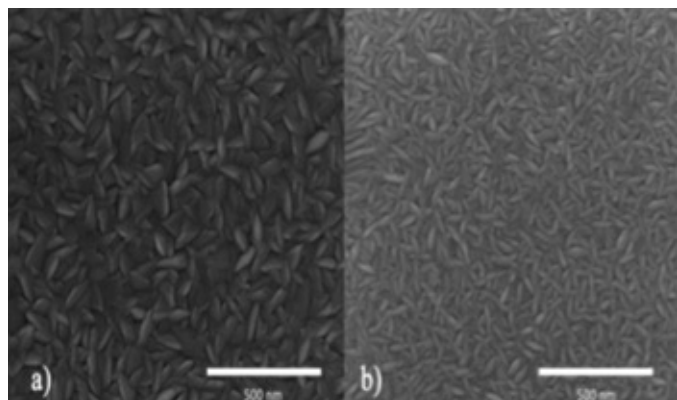


Fig. 2: SEM image of Mo with a pulse time of 1000us at a pressure of 0.44 Pa deposited with varying voltages a) 800V b) 1500V.

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Ar-Nb plasma characterization and thin film properties in HiPIMS regimes for coating of superconducting cavities

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Superconducting Radio-Frequency (SRF) accelerating cavities at cryogenic temperature are implemented at CERN to achieve high field gradients and reduce the radio-frequency losses.

SRF Crab cavities have been designed in the framework of the High Luminosity LHC project to orient the LHC particle bunches before and after the collisions in order to modify the crossing angle and obtain head-on collisions [1]. This will result in an increased luminosity. Bulk Nb Crab cavities have been developed and are currently installed and under test in the Super Proton Synchrotron (SPS).

The Wide Open Waveguide (WOW) Crab cavity, designed in the context of the Future Circular Collider study, is an alternative design featuring a thin Nb film sputtered on bulk copper [2]. The main advantages of this solution are the improved thermal stability, the reduction of the cavity frequency sensitivity to the external liquid He bath pressure variations as well as to the Lorentz force detuning, and the reduction of the material costs.

The production of the WOW Crab cavity is a complex process mostly because of its internal shape, which includes concave and convex surfaces on meter scale. This geometry requires dedicated studies to identify the optimal coating technique and cathode design to obtain a dense and defect-free Nb film to meet the target RF requirements. In this work, plasma properties (plasma density, plasma and floating potentials, ion fluxes) and thin film features in HiPIMS regimes with comparison to DCMS (with and without biased substrate), are investigated. An extensive set of plasma diagnostics is used, including Langmuir probes, Optical Emission Spectroscopy, Retarding Field Energy Analyser and Plasma Processing Monitor (PPM). In particular, the PPM allows to decouple ion mass and energy, therefore discriminating between the discharge gas (Ar) and metal (Nb) ions impinging on the substrate. Preliminary characterization of thin films obtained mimicking the sputtering process geometry that will be implemented in the real cavity is presented.

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The study of deposition method of NbN film on Cu substrate by HIPIMS

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The study of thin film cavity becomes one of the current research focuses of superconducting radio frequency (SRF) accelerator field. Among the film materials, NbN is a promising candidate material for SRF cavities because of its higher transition temperature and critical field compared with niobium.

In this dissertation, the work is mainly focused on the fabrication methods of NbN films on Cu substrates and films' properties with HIPIMS. There are diffraction peaks of NbN in the X-ray diffraction patterns. It indicates that NbN film has been prepared. The NbN film is superconductive according to results of MPMS, it shows that NbN has been reached the requirements preliminarily.

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BeW coatings deposited by hybrid HiPIMS/dcMS co-sputtering system as plasma facing material for fusion applications

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In thermonuclear fusion devices, the interaction between the particle fluxes from plasma and the surrounding surfaces results both in the reduction of the material lifetime and plasma contamination. For economic and safety reasons, plasma-facing components (PFC) must withstand harsh conditions (severe high heat flux, considerable neutron irradiation/bombardments, etc.) and they must prove a low level of dust production and nuclear fuel accumulation. These operation conditions are the most challenging parameters for any plasma facing materials (PFM) which must fulfill complex and, sometimes, contradicting requirements. Many PFMs were tested throughout the previous decades and, as a result, beryllium and tungsten seem to meet somewhat the required conditions for the ITER's walls. A comprehensible understanding of plasma-wall interactions, material migration and fuel retention in mixed materials is a big challenge for the next-step fusion device. Because the power density applied on the magnetron cathode, operating in high power pulse mode, is comparable with the one expected to be delivered to the ITER's divertor (10 MW/m²), HiPIMS system is suitable to simulate critical aspects of the plasma-wall interaction under fusion devices relevant conditions.

In this work, the so-called magnetron sputtering technique was used as model system to simulate the plasma-wall interaction in fusion devices by synthesizing Be-W layers with deuterium (D) inclusions. BeW coatings, with different Be and D content, were deposited from Be and W targets, in argon-deuterium gas mixture, using a hybrid HiPIMS/dcMS co-sputtering system. The HiPIMS mode was used to sputter the tungsten target with a fixed average power of 100 W, while the beryllium target was sputtered in dcMS mode by varying the power from 0 to 100 W. The elemental composition, structural properties and surface morphology of the obtained layers were characterized by Rutherford backscattering spectrometry (RBS), X-ray diffractometry (XRD) and scanning electron microscopy (SEM), respectively. The retained D amount in BeW layers was measured by means of thermal desorption spectroscopy (TDS).

The morphological and structural analysis revealed distinct surface morphologies and different phase composition, depending on the content of Be in the BeW layers. It was shown that the amount of retained D and thermal desorption process strongly depend on the chemical composition and microstructure of deposited mixed layers, which, in turn, can be fine-tuned through the processing parameters. It can be assumed that the high amount of D (up to 19 at.%), incorporated in BeW layers, is mainly due to HiPIMS discharge, which ensures a large flux of D ions towards the growing composite layers.

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Bipolar High Power Impulse Magnetron Sputtering: A new approach to control the metal ion flux

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Bipolar pulse High Power Impulse Magnetron Sputtering (B-HiPIMS) source have been developed and investigated in this work to control the kinetic energy of sputtered metallic ions and, therefore, the properties of the deposited coatings. The B-HiPIMS power supply consists of two HiPIMS sources, one with negative polarity and a second one with positive polarity, which can be independently controlled and synchronized through a two-channel pulse generator. The positive pulse, whose amplitude and duration can be easily controlled, can either be applied after the initial negative HiPIMS pulse (synchronized or delayed) or overlapped over it.

During the experiments, a typical set of discharge parameters, consisting of a voltage amplitude of -900 V, a pulse duration of 10 μ s and a frequency of 1 kHz was fixed for the HiPIMS supply with negative polarity, while for the power supply with positive polarity, the voltage value was varied between 0 and +200 V, the pulse duration was varied between 2 and 50 μ s, while the delay between the ignition time of negative and positive pulses was varied between 5 and 50 μ s.

Mass spectroscopy was used to analyse the energy of the ion flux during B-HiPIMS of a copper (Cu) target, in argon (Ar) atmosphere. The ion energy distribution functions (IEDFs) were recorded in the time-averaged mode, for Ar⁺ and Cu⁺ ions. The influence of the positive pulse configuration on both kinetic energy of plasma ions and properties of the deposited coatings was investigated. Structural properties of the coatings deposited in monopolar HiPIMS and B-HiPIMS, with different values of positive voltage, were investigated by Rutherford backscattering spectrometry (RBS), X-ray diffractometry (XRD) and scanning electron microscopy (SEM).

The ion energy distribution function, ion flux and mean energy of Ar⁺ and Cu⁺ ions strongly depend on the positive pulse's characteristics: amplitude, delay and duration. Both ion flux and mean energy of metal ions can be fine-tuned in a wide range by modifying the pulse configuration. As compared to monopolar HiPIMS, during B-HiPIMS (positive voltage pulse of 200 V, pulse duration of 10 μ s and delay time of 9 μ s), the high-energy tail of Cu⁺ IEDF extends from 100 eV to 300 eV, the mean energy increases from 7 eV to 150 eV, while the ion flux increases 4 times. The influence of the positive pulse configuration on the kinetic energy of Ar⁺ ions is even more significant: the mean energy increases from 1 eV to 190 eV, while the ion flux increases 10 times. In both HiPIMS operating regimes, mono- and bipolar, the deposited coatings are highly textured. Although in B-HiPIMS there is no net increase in the deposition rate as compared to the monopolar regime, the coatings deposited by B-HiPIMS seem to be denser, smoother and of higher crystallinity. These improvements in film properties may be attributed to the intense and energetic ion bombardment during the deposition process of the coatings.

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Chromium target composition in reactive high-power impulse magnetron sputtering characterized by in-vacuum X-ray photoelectron spectroscopy

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High power impulse magnetron sputtering (HiPIMS) has established itself as one of the premier methods for depositing high quality hard coatings. Reactive gases can be added to the discharge to produce hard ceramic coatings. These gases can react with the target surface which is called “target poisoning”. It has often been claimed that target poisoning is accompanied by a strong change of the current waveform but direct experimental verification is still needed.

This was addressed by connecting spatially resolved X-ray photoelectron spectroscopy (XPS) measurements with measurements of the current waveform. The XPS characterization was performed after an in-vacuum transfer of the target to avoid any oxidation or contamination.

A chromium target was used for the discharge. The Ar/O₂ mixture, the input power and the frequency were varied to evaluate the racetrack oxidation state in different discharge regimes.

The transition from poisoned to metal mode by increasing the power can be achieved using a frequency of 20 Hz. This transition can as well be seen in the current shape of the discharge which converges to its non-reactive form. This is a first approach to investigate the assumed correlation.

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Control of voltage and current peak shape for reactive HIPIMS deposition

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The dense and high quality HIPIMS coatings draw attention of many industries as a mean to improve the quality of wear protective, decorative or biomedical coatings as well as for photovoltaics or integrated circuit production. At the beginning, a low deposition rate and high arcing rate in reactive processes have limited its application on industrial scale. However, a constant development of the HIPIMS technology brought better understanding of the HIPIMS processes as well as expanded the number of industrial users.

Successful implementation of a new technology into mass production requires an in-depth understanding of the technology background as well as of its functionalities previously unknown and inaccessible with older solutions. Therefore this paper is focused on one of the biggest fundamental change in the HIPIMS pulse generation, namely the voltage and current shape control and a change from unregulated release of the capacitor bank energy into plasma to precisely regulated HIPIMS pulse peak currents with amplitude as high as 2000 A.

First the influence of fast current rise in a quasi-rectangular HIPIMS pulse on the plasma development will be discussed. The discussion will be followed by an analysis of a quasi-rectangular voltage and current pulse on process-related factors. Thus, it will be showed that application of quasi-rectangular HIPIMS pulse does not lead to increased probability of arc formation in metallic and reactive sputtering modes. Moreover application of controlled voltage and current peak shape in average power regulation mode will be demonstrated as an effective regulation method for reactive processes. In addition the reduction of arc rate leading to a higher deposition rate will be analysed both for the quasi-rectangular HIPIMS pulse and average power regulation mode.

Finally different solutions of HIPIMS integration into deposition process of TiN coatings with a defined adhesion, hardness and colour requirements will be discussed.

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Industrial system solution approach utilizing HIPIMS for improved process optimization: a Ti-Cr-based anticorrosion coating case study

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As the magnetron sputtering deposition of functional coatings is a common production step used for variety of high-tech and daily-use products, a lot of manufacturers invested much effort into the optimization of this technological step. The optimization itself, is commonly focused on process metrics such as its throughput or effectively judged by the amount of faulty products from one process batch.

Plasma power supply is one of the key components required for magnetron sputtering deposition and in many cases a system used for coating deposition uses multiple magnetron sources. Furthermore, in industrial scale plasma deposition single and dual magnetron arrangement are used in a form of planar as well as rotatable targets. The experience gathered in the laboratory scale demonstrated dramatical improvement of the physical quality of sputter deposited coatings by High Power Impulse Magnetron Sputtering (HIPIMS). To take the benefit of improved coating parameters with HIPIMS the technology must not only be scalable to standard industrial scale but also allow its application for different target configuration, including the dual rotatable systems used i.e. in large area coating deposition.

This contribution focuses on a novel approach for HIPIMS application in dual magnetron sputtering systems. First, a brief overview of industrial applications of dual magnetron arrangements will be given. Based on the challenges known i.e. in the large area coating, where MF and Bipolar power supplies are used as standard plasma generators, the possible field of application for a Bipolar-HIPIMS (B-HIPIMS) units will be introduced. As an industrial example of a successful application of B-HIPIMS the processing condition and physical properties of a Ti-Cr-based anticorrosion coating deposited by mixed Bipolar and B-HIPIMS technology will be discussed. Thus, the operational stability and process repeatability of B-HIPIMS unit working on dual rotary magnetrons will be analyzed in details. A special attention will be devoted to the fulfillment of strict requirements for delivery of average power and peak current up to 1000 A in order to meet the criteria of a HIPIMS-like discharge on dual targets with 1.2 m length each. Next, the Bipolar and B-HIPIMS mixing will be shown to provide superior adhesion and mechanical properties of the Ti-Cr-based coating despite a slightly reduced deposition rate. Finally, analysis of the full system solution approach will be given, where magnetron and bias power supplies are synchronized together for further improvement of coating quality and production yield.

The discussion will be concluded with a recent results of B-HIPIMS qualification for large area coating applications, where dual target arrangement with target length of 3.75 m are used as a standard.

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Advantages associated with applying a Positive Pulse option to a HIPIMS power supply

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HIPIMS is an ionized PVC technique that produces a high density, high performance films. The extreme power densities in HIPIMS create a higher ionized plasma that creates a very high energy of material being deposited onto the substrate.

Many advanced techniques have been found to further enhance the quality of HIPIMS films, creating more ideal process and applications for utilizing this technique.

We will show advantages of integrating a positive “kick” pulse into a HIPIMS application. The “kick” pulse is an ideal feature for reactive sputtering applications due to its ability to carry out the HIPIMS plasma for extended period of time, minimizing the disapperating anode effect and repelling metal ions from the plasma toward the substrate resulting in higher sputtering rates.

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Gas rarefaction in HiPIMS – comparison of a particle simulation and volume-averaged models

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A fully three-dimensional simulation of transport of neutral atoms in a high power impulse magnetron sputtering discharge was developed using the Direct Simulation Monte Carlo (DSMC) method since the transport of material in the discharge is of high importance as far as the film quality and the deposition rate are considered. In this work, we simulate the time-dependent evolution of gas and target material densities in the discharge during sputtering pulses, especially the dynamics of gas rarefaction during the pulse and gas refill during the pulse-off time. The simulation geometry is sufficiently large to allow for gas expansion beyond the region between the target and substrate.

Simulations with the argon pressure of 1 Pa and three target materials with significantly different masses (Zr, Al and C) and various sputtering currents were carried out for pulses of 500 Hz repetition frequency and 10% duty cycle. In all cases, steady state of the argon density is not reached during the 200 μ s sputtering pulse. During the pulse-off time, argon reaches equilibrium with homogeneous density distribution in the discharge chamber approximately 1 ms after the pulse end. The argon density decreases to 50% of its initial value for the current density of sputtered Al atoms of around 0.5 Acm⁻². For Zr and C, the minimum argon density is 43% and 57%, respectively, at the same current densit. The dependence on the mass of the target material is found to be rather weak.

Volume-averaged models have been used recently to make simplified predictions of the HiPIMS plasma parameters. The calculation of the neutral gas transport is, however, problematic with the volume-averaged approach. We present two representative volume-averaged models of the gas and target material density in front of the target and compare results of these models with the DSMC simulation. We discuss the strengths and weaknesses of the volume-averaged models and point out the critical parameters influencing their accuracy.

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Novel HIPIMS deposited nanostructured CrN/NbN coatings for environmental protection of steam turbine components.

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To increase efficiency, modern steam plants are pushing their operational regime from super-critical (600°C/300 bar) to ultra- super-critical (740/760°C/350 bar) stretching existing turbine materials to their limits. The focus is on new generation functional materials and technologies which complement the inherent properties of existing materials.

Current work proposes a novel High Power Impulse Magnetron Sputtering (HIPIMS) deposition technology, for the first time, for deposition of a ceramic based CrN/NbN coating with a nanoscale multilayer structure (bi-layer thickness $\Delta = 1.9$ nm) with superior adhesion ($L_{C_2} = 80$ N) to protect low Chromium P92 steel widely used in steam power plants. Thermodynamic calculations predict the equilibrium phases and aggressive gaseous compounds generated by the interaction of steam with the coating. CrN/NbN coated P92 steel samples oxidised at 600°C in a high pressure (50 bar) 100% steam atmosphere for up to 1000 h reveal the coating's superior oxidation resistance and protective mechanisms, especially against the detrimental effect of Hydrogen. High temperature (650°C) Tensile Strength, Low Cycle Fatigue and Creep tests confirm that, unlike other state-of-the-art PVD technologies, HIPIMS is not detrimental to the mechanical properties of the substrate material. Water droplet erosion tests confirm no measurable weight loss after 2.4×10^6 impacts.

Keywords: hipims, CrN/NbN, nanoscale multilayers, steam oxidation resistance, water droplet erosion resistance.

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Coated Shoulders for Friction Stir Welding of Aluminium Alloys

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Friction stir welding is a cold joining process for metal alloys which involves the plunging of a probe into the joint and rotating it to mechanically stir the two parts together. Recently a stationary shoulder technique has allowed for a smoother finish of the weld. The challenge remains to eliminate post-processing steps altogether. Surface coating of the probe and the shoulder have been rarely successful due to the aggressive nature of the work piece material when it is brought up to temperatures close to its melting point during the operation. The coatings must be well adherent in order to withstand probe deformation and prevent chemical reactions with the workpiece material to avoid buildup. The stationary shoulder friction stir welding of Aluminium AA7075-T6 alloy has been used to compare HIPIMS-deposited TiAlN/VN coatings with amorphous DLC by PECVD, AlTiN by cathodic arc evaporation and TiBCN by CVD methods. Sliding wear tests at temperatures between room and 350°C revealed a stable friction and wear coefficient for the TiAlN/VN coating and increase in friction for the aDLC. Energy dispersive X ray analysis was used to study the composition of the buildup material on worn shoulders and wear tracks on HSS coupons. Welding tests showed that the TiAlN/VN prevented the buildup of workpiece material, whereas the aDLC sustained a buildup due to softening at temperatures in excess of 350°C when the coating graphitises as observed by Raman spectroscopy. The best-performing coatings for the shoulder were those with low affinity to Aluminium and low coefficient of friction -TiAlN/VN and aDLC. Both coatings provided an excellent weld quality with a surface finish smoother than that of the original materials.

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Properties of spokes observed with a linear magnetron

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Abstract

Spoke evolution in magnetrons has been extensively studied over the last five years. Especially powerful were fast camera images since they convey multidimensional information on the magnetron plasma. Among fast cameras, streak cameras are of special interest since their Intensity(x,t) images deliver information with high spatial (better than 1 mm in one dimension) and time resolution (down to ns). Since the spatial information is in one direction, a linear magnetron offers the unique opportunity to align the (linear) slit of a streak camera with a linear section of the racetrack [1, 2]. Streak images contains lots of information including the spoke travel direction and velocity, the spoke size and intensity, spoke substructure, if present, and the evolution of a spoke relative to its neighboring spokes. For example, some images show self-organized patterns, while others indicate a more chaotic behavior. Streak images also show somewhat unexpected events, like one spoke “catching up” and merging with the preceding spoke, or one spoke splitting into two having different velocity. Clearly, this experimental study shows that our understanding of these complicated events is rather incomplete, and only reasonable, qualitative explanations are provided. Explanations involve the presence of atoms to be ionized, stemming from the background gas, sputtering, and target outgassing, and the availability of sufficiently energetic (“hot”) electrons, which are primarily energized in the potential structure of the spokes themselves.

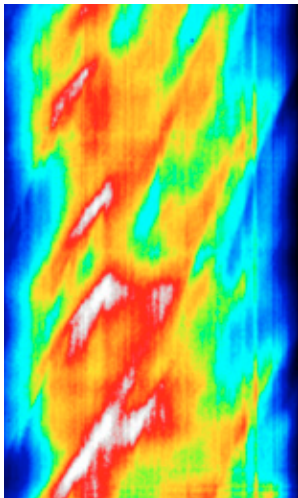


Fig. 1:

Example of a streak image, with light intensity expressed by false color (horizontal 20 cm, vertical 20 μ s, with time increase pointing down, Ti target, in 0.4 Pa Ar, at about 600 A, 300 pulses with 20 Hz repetition frequency.

Acknowledgements

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Optimization of magnetic field configuration for Titanium Nitride deposition using HIPIMS

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The particular characteristic of high power impulse magnetron sputtering (HIPIMS), compared to conventional dc magnetron sputtering (dcMS), is the high ionization fraction of the sputtered material, offering interesting film properties such as higher density. In this study, the effect of varying the magnetic flux density B from 55 mT to 103 mT on the deposition rate of titanium nitride for the HIPIMS process was investigated. The effect for dcMS was also studied for comparison. The results show a decrease of deposition rate with increasing B , by 35% in the case of HIPIMS and by 19% in the case of dcMS. The effect on coating morphology showed an evolution from featureless morphology to dense columnar with increasing B in the case of HIPIMS. In the case of dcMS, the density of the columnar structure seems to increase relatively with increasing B . The X-ray diffraction pattern indicates, in the case of HIPIMS, a fine-grained microstructure and an increasing preferential (111) orientation as B increases. For dcMS, the XRD indicates larger crystals and there is a gradual change from (200) to (111) as B increases.

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Probing the Electron Density of Spokes Using Target Inserts

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At high discharge currents, the plasma emission of a HiPIMS discharge is not homogeneous but is instead organized into distinct zones of high plasma emission, which rotate in ExB direction a few mm above the target surface. These so-called “spokes” have received a lot of attention in the recent years. The strong emission indicates an elevated electron density. However, it would disturb the plasma considerably to position a probe in the vicinity of the target. Therefore, no direct measurement of the electron density inside the spokes has been performed. In this contribution, small electrically isolated inserts in the target surface were used to probe the local current density. Simple sheath theory was then applied to derive the electron density at the sheath edge. The measured electron density was in the order of 10^{19} m^{-3} and scaled linearly with discharge current. The electron density rose by about 50% when a spoke was present.

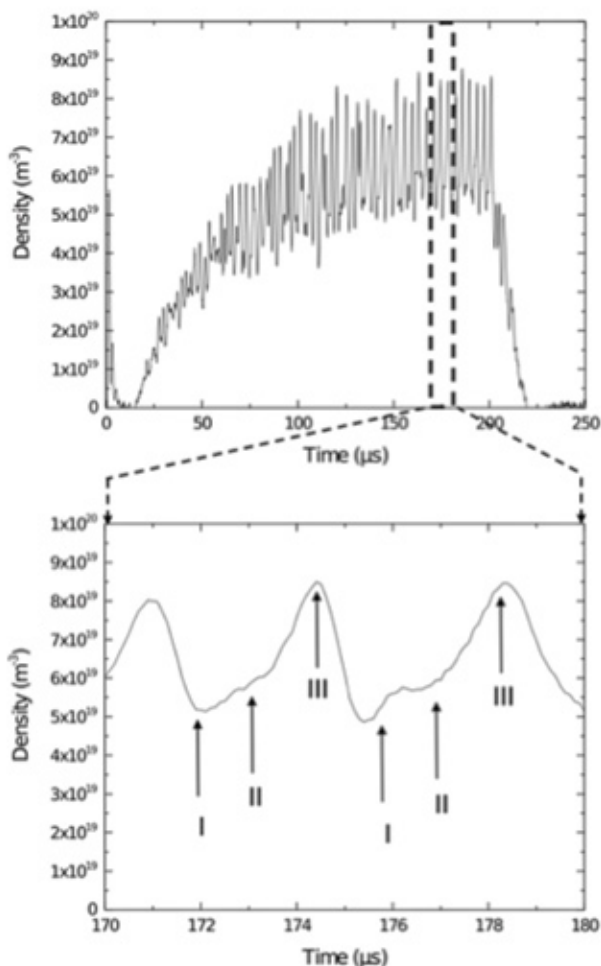


Fig. 1:

Electron density at the sheath edge during the discharge pulse. Fluctuations in density are caused by moving Spokes.

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Effect of pulse power on controlled reactive HiPIMS deposition of ZrO_2 films examined by an optical emission spectroscopy

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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 feedback 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.

Here, we report on the results of the optical emission spectroscopy with a temporal resolution of 320ns carried out near the target and in the plasma bulk during the deposition of densified ZrO_2 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, diameter of 100mm). Oxygen was admitted directly into a high-density plasma in front of the target via two corundum conduits (located 25mm from the target) with the inlets (diameter of 1mm) directed to the substrate. The actual O_2 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 O_2 partial pressure in the discharge. The repetition frequency of the voltage pulses with a duration of $t_{\text{on}} = 50\mu\text{s}$ and $t_{\text{on}} = 200\mu\text{s}$ was 500Hz at the deposition-averaged target power density of $52\text{--}53\text{Wcm}^{-2}$ (peak target power density was 3.1kWcm^{-2} at $t_{\text{on}} = 50\mu\text{s}$ and 1.1kWcm^{-2} at $t_{\text{on}} = 200\mu\text{s}$). The Ar partial pressure was 2Pa. Under these conditions, the deposition rate of ZrO_2 films up to 120nm/min was achieved at $t_{\text{on}} = 200\mu\text{s}$ on a floating substrate 100mm from the target.

From time evolutions of the excited-state populations for the chosen atoms (Zr, Ar and O) and ions (Zr^+ , Zr^{2+} , 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 were derived. It was found that approximately four times higher value of the average target power density in a pulse ($1700\text{--}2100\text{Wcm}^{-2}$ at $t_{\text{on}} = 50\mu\text{s}$ compared to $370\text{--}540\text{Wcm}^{-2}$ at $t_{\text{on}} = 200\mu\text{s}$) leads to more intense Ar atom density reduction near the target due to a combined effect of a strong rarefaction and ionization of the Ar atoms. In addition, a significantly higher ionization degree of sputtered Zr atoms was observed near the target. This increase in the ionization degree of the Zr atoms was not observed in the plasma bulk indicating that a high number of the produced Zr^+ and Zr^{2+} ions are attracted back to the target under these conditions. A much higher O atom density found in the plasma bulk at $t_{\text{on}} = 50\mu\text{s}$ indicates a higher dissociation degree of O_2 molecules in this case. Contrary to what was found at $t_{\text{on}} = 200\mu\text{s}$, a higher content of oxygen in the discharge during the O_2 pulses at $t_{\text{on}} = 50\mu\text{s}$ did not induce a decrease in the excitation temperature following trends in the electron temperature.

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Controlling the polymorphic composition of TiO₂ thin films by modifying the target surface conditions using HiPIMS deposition parameters

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The polymorphic composition and crystallinity are important parameters, influencing the optical properties of thin TiO₂ films. In this work, the influence of the target poisoning on the polymorphic composition, texture and crystallinity of stoichiometric TiO₂ thin films using high-power impulse magnetron sputtering (HiPIMS) deposition was investigated. For a pulse duration of 50 μs, the deposition rate was very low (~0.5 nm/min), with the films being sub-stoichiometric and mixed-phase in nature. The deposition rate started to increase quadratically up to 3.4 nm/min with the increase in pulse duration up to 500 μs, whilst the mixed-phase nature of the TiO₂ films evolved starting from anatase to rutile, when the maximum pulse duration was applied. The application of 100 V negative substrate bias reduces the TiO₂ rutile formation temperature threshold by 100 °C, while the anatase phase was predominant when the substrate was self-biased (-30 V) at the same deposition temperature. Appropriate selection of pulse duration increases the deposition rate, enables to choose the phase between anatase and rutile, and facilitates a stable reactive deposition of stoichiometric TiO₂ rutile films with excellent optical properties. The target surface changes smoothly from a poisoned state, at short pulse duration, to a quasi-metallic state at long pulse duration. As the operation transitions from Ar driven sputtering to metal driven sputtering, the oxide layer on the target surface changes and decreases the energetic oxygen ion bombardment on the film surface whilst increasing the Ti/Ar flux ratio. The variations in film morphology and polymorphic composition can be attributed to the change in chemical condition of the target surface. Finally, we show that by tuning the pulse characteristics, alongside other HiPIMS parameters, the refractive indices of the TiO₂ films can be controlled with little variance in the absorption coefficients which are important for the fabrication of optical multilayer stacks.

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Low-temperature deposition of thermochromic VO₂ films on glass and kapton using reactive deep oscillation magnetron sputtering

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A modified version of HiPIMS, called Deep Oscillation Magnetron Sputtering, with a pulsed O₂ flow control and to-substrate O₂ injection into a high-density plasma in front of the sputtered vanadium target was used for low-temperature (330 °C) deposition of thermochromic VO₂ films onto conventional soda-lime glass (1 mm thick) and flexible kapton polyimide foil (25 μm thick) substrates without any substrate bias voltage and without any interlayer.

The depositions were performed using a strongly unbalanced magnetron with a planar vanadium target of 100 mm diameter in argon-oxygen gas mixtures at the argon pressure of 1 Pa. Voltage macropulses, composed of 10 voltage micropulses (pulse-on time of 22 μs and pulse-off time of 28 μs), with a total length of 500 μs and repetition frequency of 400 Hz were used for all depositions with a maximum target power density of up to 1130 Wcm⁻² during pulses at a deposition-averaged target power density of 21 Wcm⁻².

A high modulation of the transmittance at 2500 nm (even between 77% and 17% for VO₂ films on the kapton substrate) was achieved for the VO₂ films on the glass and kapton substrates at the transition temperatures of 57-64 °C.

This low-temperature magnetron sputter technique is of key importance for compatible fabrication of thermochromic VO₂-based multilayer coatings for smart windows and smart radiator devices (spacecrafts) applications requiring enhanced luminous transmittance and solar transmittance modulation at a decreased transition temperature.

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Niobium coatings onto complex accelerating structures by HiPIMS

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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 based on SRF cavities using copper coated with a niobium thin film [1]. With respect to their counterparts made out of bulk niobium such an approach allows the reduction of the fabrication costs and the copper thermal conductivity suppresses the possibility of having a quench event .

We will show in a the first time that niobium layers coated by HiPIMS have already given promising indications of enhanced performances compared to layers elaborated by standard Direct Current Magnetron Sputtering (DCMS) [2].

In addition to the RF performances, HiPIMS technique could also enable the possibility to coat very complex shapes such as low beta cavities keeping a dense layer all over the substrate surface. In such geometries in DCMS most of the Nb atoms impinge the surface at grazing angle and lower the density of the coating. The latter has shown to be responsible for depressed RF performances [3, 4]. In contrast HiPIMS could lead to large incidence angles and densification.

To achieve such a result we will show that two conditions need to be satisfied. First of all the layer densification can only be obtained by applying a negative bias onto the substrate in order to attract the metallic ions on its surface. Second, the ionization region has to be properly tuned to achieve a better film thickness uniformity on the surface. We will describe how such a result can be obtained by carefully designing the magnetic assembly of the magnetron source.

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Deposition of Niobium for Superconducting Accelerator on Copper by High Power Impulse Magnetron Sputtering

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The challenge in thin film techniques applied in RF superconducting accelerator is to reproduce and even go beyond the performance of bulk Nb. HiPIMS, as a kind of promising emerging ionized-PVD coating technique, is used to increase the performance of niobium films. A new HiPIMS power supply used for niobium deposition were developed by Peking University and China Academy of Engineering Physics. The maximum pulse output power is 70kW and the pulse length is 200us. Pre-ionization pulse and substrate bias pulse were designed and appended in this HiPIMS power supply. Niobium films were deposited on copper samples by dcMS and HiPIMS. Superconducting performance and films properties of different niobium samples were compared.

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Laser annealing for niobium film on copper deposited by HiPIMS

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Niobium thin film coated copper cavities are a promising alternative to bulk niobium cavities for RF superconducting accelerators. The challenges are improving the surface superconducting performance and reducing defects of the coating film, which can be greatly solved by laser annealing. Short pulse laser can produce a sharp step temperature field in one-pulse time, and this process occurs on the film thickness measure which anneals the thin film with substrate protected. Surface appearance and other properties of dcMS and HiPIMS samples were compared after annealing. Because of the higher density and compactness of HiPIMS samples, cracks decreased a lot and better recrystallization happened according to the results.

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Dual-HiPIMS system as source of fusion related W-Al composite layers having helium and deuterium inclusions

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Material erosion, migration and re-deposition are serious concerns for future nuclear fusion reactors. Materials eroded from the first wall and the divertor area can heavily contaminate the fusion plasma, leading to energy losses and plasma instabilities, such as edge localized modes (ELMs), which can seriously damage the reactor. Another concern regards the in-vessel tritium (T) inventory which has an imposed limit of 700 g for the International Thermonuclear Experimental Reactor (ITER) due to safety regulation. The largest part of the tritium inventory is expected to be retained in co-deposited layers, which justifies production and study of co-deposited layers with helium (He) and deuterium (D₂) inclusions; He as the main product of the fusion reaction and deuterium as surrogate for tritium. The ITER vessel will be composed of beryllium (Be) and tungsten (W) but, due to safety regulations regarding the handling of T and Be, their use in experimental facilities is limited. Aluminum (Al) is similar to Be in oxide and deuteride formation. This qualifies it as surrogate for Be in experimental studies. The present experiments used the high power impulse magnetron sputtering (HiPIMS) as deposition technique since it was successfully implemented to obtain and study fusion related coatings [1,2].

Mass spectrometry measurements have been performed for plasma characterization in a dual-HiPIMS discharge (W and Al targets), in Ar-He and Ar-D₂ gas mixtures. The total ion flux and its composition strongly depend on the target material, gas mixture composition and the input power delivered to each target. It increases with the electrical power applied to each target as well as with He/Ar or D₂/Ar ratio. During W and Al co-sputtering process, in Ar-He gas mixture, for the same average power applied to each target, the ion flux is dominated by Al⁺ ions, regardless the gas composition. In Ar-D₂ gas mixture, for an average power applied to the Al target lower than 60 W, the ion flux is dominated by D⁻ ions, while for higher power values, the ion flux is dominated by Al⁺ ions.

The influence of the average power on the microstructure, chemical composition and deuterium retention of the films was investigated by SEM, XRD and GDOES depth profile measurements. GDOES profiles indicate the presence of a large amount of deuterium in the W-Al layers, with a maximum value of 21% and a mean value (estimated over the entire thickness of the layer) up to 15%. Depth profiles indicate that the D distribution in the W-Al layers is related to the W in-depth concentration. Experimental results show that the main mechanism responsible for the D retention in the W-Al layers is the intense and energetic bombardment of the growing film with D⁻ ions. Therefore, D⁻ ions were investigated in detail during standard and dual-HiPIMS with W and Al targets. The energy distribution functions of D⁻ ions show a maximum around 1 eV and a very high energy tail which extends towards 500 eV. During standard HiPIMS operation, the D⁻ ion flux linearly increases with the input power on the W target and with the increase of D₂/(Ar-D₂) mass flow ratio. During dual-HiPIMS operation, at high D₂/(Ar-D₂) mass flow ratio (50 and 75%), the D⁻ ion flux tends to decrease as the input power on the Al target increases.

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First HiPIMS activities at Politecnico di Milano

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In the present work the first activities of a HiPIMS apparatus newly installed at Politecnico di Milano are presented. Exploiting the features of this deposition technique compact metallic and carbon coatings are studied in the framework of the design and production of target systems to be used in laser driven ion acceleration experiments.

Within the “ENSURE” ERC project a new HiPIMS deposition system has been recently installed at Politecnico di Milano. It consists in a dual confocal magnetron sputtering system equipped with 3-inch cathodes. High power pulses are provided by a Melec SIPP 2000 Dual DC pulse power controller. Exploiting the features of this controller it is possible to grow coatings in several modalities: bipolar, unipolar, unipolar or bipolar + pulsed bias. It is also possible to superimpose HiPIMS and MF pulses with the aim of sustaining the plasma between the HiPIMS pulses thus improving the deposition process.

The aim of the first experimental campaigns is to find the best deposition parameters to grow compact micrometer thick coatings both of carbon and metal (e.g. Ti) to be used as free-standing layer. This layers with the addition of an ultra-low-density carbon foam deposited by Pulsed Laser Deposition (PLD) will be the target materials used in laser driven ion acceleration experiments [1].

PLD is a very versatile technique, compared to standard magnetron sputtering, and allows the deposition of compact metallic films with a crystalline size from hundreds of nanometers down to the amorphous regime without significantly affect film morphology [2]. But on the other side, this deposition technique suffers of two main issues that are: small deposition areas and ejection of liquid droplets from the target. Thanks to its high energy and pulsed deposition regime HiPIMS could mimic PLD deposits without suffer the PLD drawbacks. The first comparison between metallic coatings produced by HiPIMS and high energy PLD will be presented.

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Influence of spokes on the ionized metal flux fraction in chromium high power impulse magnetron sputtering

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A quartz crystal microbalance (QCM) is combined with an ion repelling grid system (IReGS) to investigate the deposition rate and the ionised metal flux fraction (IMFF) of a chromium HiPIMS discharge in dependence of the peak power density. The measurements are supported by ICCD camera images to analyse the appearance of spokes. Energy resolved ion mass spectrometry is used to determine the ion energy distribution function (IEDF) and the ion flux. The deposition rate decreases with increasing peak power density, while the IMFF and the ion flux increase. At a peak power density of 0.25 kW cm^{-2} all these trends slow down. These changes in the trends correlate with the appearance of spokes observed in the ICCD images, indicating that the formation of spokes counteracts the return effect in HiPIMS. With the average ion energy calculated from the IEDF the energy per deposited ion is calculated and a peak power density of 0.5 kW cm^{-2} is found to be the optimum for a high deposition rate and a sufficient high energy per deposited ion at the same time.

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HIPIMS for pretreatment and coating of plastics

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Metallization of plastic parts today is mainly realized using electroplating. Within the European Union the use of chromium VI will be restricted by 2017 following the REACH directive. This besides other aspects like environmental friendliness is driving the development of alternatives. Using PVD mainly evaporation is used. The drawback is sometimes weak adhesion. Furthermore laquers or interface coatings have to be applied before metallization.

Using ionized sputtering like high power impulse magnetron sputtering HIPIMS opens new horizons for cost effective, environmental friendly plastic metallization with excellent adhesion. In the best case, excellent adhesion is realized without pre-treatment by direct metallization. First studies showed that even PMMA can be directly metallized by HIPIMS Plasma. Additionally cheap plastics like PP could be used, also for thick electroplated metallization, if a full coverage with an adhering interface layer is provided. Therefore, besides the study of direct metallization of different plastics the potential of a HIPIMS plasma pre-treatment is presented.

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Effect of chamber pressure on defect generation and their influence on corrosion and tribological properties of HIPIMS deposited CrN/NbN Coatings

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It was reported that compared to state-of-the-art technologies, High Power Impulse Magnetron Sputtering produces very dense and droplet free coatings due to the high plasma density and ionisation rate. However, thorough investigation of the coating morphology by Scanning Electron Microscopy, optical microscopy and other surface analysis methods revealed the existence of various types of coating defects.

The paper reports the influence of chamber pressure in particular on defect formation in CrN/NbN nanoscale multilayer coatings. The coatings were deposited using a combined HIPIMS/UBM technique by varying the total chamber pressure from 0.2 Pa to 1 Pa. Four types of defects were identified, namely, nodular, open void, cone-like and pinhole. Defect density calculations showed that the coating produced at the lowest pressure, 0.2 Pa, had the lowest defect density of 0.84 %.

As expected coating corrosion properties were improved linearly with decreasing defects. Potentiodynamic polarisation corrosion studies revealed that in the potential range of - 300 mV to + 300 mV, the current density decreased with decreasing defect density (from 5.96 % to 0.84 %). In contrast, pin-on-disk tribology tests at room temperature demonstrated that the tribological properties of the coatings deposited at different chamber pressures were dependent on the crystallographic orientations and on the nature of the oxides formed at the tribological contact. Coatings with (200) crystallographic orientation had lower wear rates ($\sim 1.6 \times 10^{-15} \text{ m}^3 \text{ N}^{-1} \text{ m}^{-1}$) whereas coating with (111) crystallographic orientation had the highest wear rates ($2.6 \times 10^{-15} \text{ m}^3 \text{ N}^{-1} \text{ m}^{-1}$). Friction properties were influenced by the tribolayer formed during the tribological tests.

However, for the coatings deposited at same chamber pressure of 0.35 Pa but with different defect densities, the friction behaviour was directly influenced by the coating defects. The friction co-efficient (μ) decreased by a factor of two from 0.48 to 0.25 when the defect density decreased from 3.18 % to 1.37 %.

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