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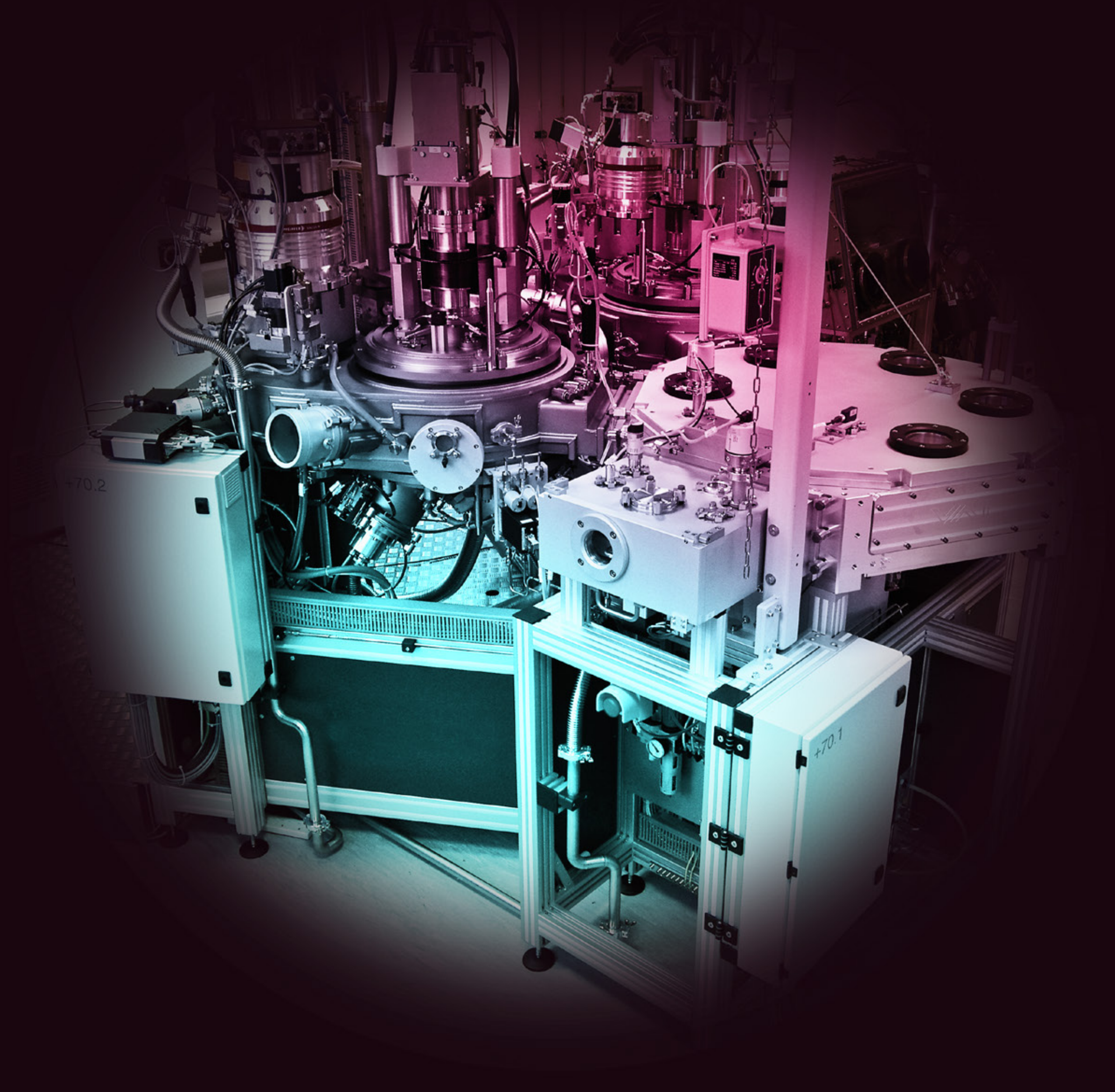
# Seventh International Conference on HIPIMS
# *Abstract Book*



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# Contents

# HIPIMS ITO from ceramic and metallic rotating cathodes

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HIPIMS is a deposition process, which produces high quality films from plasmas with a high content of ionized sputtered species. For the fabrication of ITO films, a ceramic target is typically used. Ceramic targets are more expensive than metallic ones, provide low deposition rates, and are a source of energetic negative oxygen ions which can cause damage and degradation of film properties. Since HIPIMS makes use of high voltage pulses, problems due to highly energetic negative ions are a concern. A reactive process using metallic targets will reduce the amount and energy of negative oxygen ions. For a reactive HIPIMS process on lab scale, numerous advantages like, better coverage of complex-shaped surfaces, higher deposition rate compared to non-reactive mode, room temperature deposition, decreased lateral resistivity, and decreased surface roughness are reported. Nevertheless, up-scaling the process to industrial scale still needs a reliable control of the oxygen flow for large area rotatable targets. In this work, a reactive HIPIMS process for deposition of ITO from a 0.6 m long rotatable target was investigated. A suitable closed-loop control of the oxygen flow was developed, making use of optical emission spectroscopy. The oxygen line emission was used as feedback signal. A comparison between the reactive process and a non-reactive process from a ceramic target is presented in terms of the main features of the processes and the respective deposition rates. A comparison between films deposited from ceramic and metallic targets is shown in terms of electrical and optical properties.

**Keywords**: HIPIMS, ITO, optical emission, rotatable, reactive, oxygen.

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# HiPIMS deposition of tungsten trioxide thin films

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Tungsten oxide is well known for its electrochromic and photocatalytic properties.

The first series of thin films of $WO_3$ were deposited using a single magnetron which was situated directly under the sample holder. Process gas (Ar) at a flow rate of 20 sccm and reactive gas ($O_2$) at a flow rate of 8 sccm were let in the chamber. As a power supply for the magnetron an ADL Gmbh. DC unit with a MELEC Gmbh. SIPP2000USB plasma DC pulse power controller was used. During the cycles the pulse shape was monitored using an oscilloscope and OES monitoring was conducted for further studies. Duty cycles of the waveforms were starting from 0.3% to 5%.

The second series of $WO_3$ thin film samples were sputtered while heating substrates. Those samples which were sputtered at the temperature of 300°C (ID Nr. 1160, 1161, 1162) clearly show crystalline structure in the XRD spectra. The sample 1163 shows only one narrow and weak intensity XRD peak, which means that sputtering parameters of this sample are borderline between roentgen-amorphous and crystalline atomic structures.

In Raman spectra all samples show distinctive peaks at 700 and 800 cm-1 (Fig.1), which is typical for $WO_3$ monoclinic lattice. Halfwidth of these peaks and their position are dependable on the size of the crystallites which will be studied further.

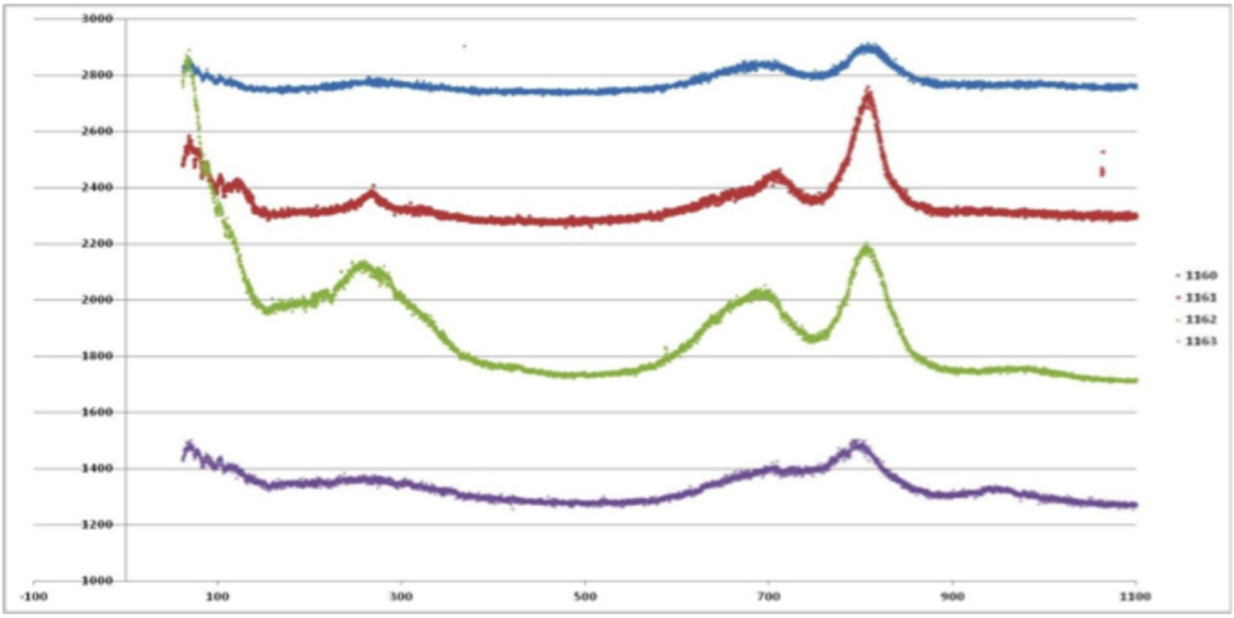


**Fig. 1:**

Raman spectra for samples (from top to bottom) 1160, 1161, 1162, 1163

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# HiPIMS plasma diagnostic and low temperature deposition of photo-active titania thin films in an industrial-scale rig

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In the field of PVD techniques, the High Power Impulse Magnetron Sputtering (HiPIMS) process [1] is one of the newest and most promising development for low temperature depositions. Its understanding is still intensely investigated and reported in numerous articles and reviews every year [3]. Indeed, a better knowledge of the processes occurring within the plasma with precision is fundamental for its use to be effective during thin films depositions.

The study of the process is more complex than Direct Current Magnetron Sputtering (DCMS) and mid-frequency pulsed magnetron sputtering - due to a longer list of parameters and phenomena occurring - and need to be conducted with caution [2]. In particular, for the case of reactive sputtering: it has been demonstrated several times that the reactive gas has a huge impact on the plasma behaviour. It is also a very sensitive process. The hysteresis behaviour widely described in the literature [4] [5] implies that the reactive gas content should be regulated with precision along time during the deposition duration to keep the plasma stoichiometry constant. To do so, the Speedflo™ gas controller for HiPIMS systems [4] has been used for the work presented here. This gas flow controller measures the optical signal emitted by the energetic species of the plasma and regulates the reactive gas flux in order to keep the stoichiometry to a chosen value. The effectiveness of the device has been confirmed and used to conduct a more precise study on the influence of the oxygen content while sputtering a titanium target.

Ti and Ti-O discharges have been analysed in terms of deposition rates, thermal load at the substrate position and pulse shapes modifications while varying the whole range of conditions: pressure, voltage, pulse width, frequency and oxygen content. A stable process envelope has been determined for the rig used in this study. Within this envelope, each parameter's influence has been studied and understood for both metallic and stoichiometric discharges. The consequences of these variations have then been observed for film growth processes. Several analysis techniques have been used (SEM, EDX, Raman, XRD, etc…) to determine which conditions resulted in a crystalline growth of the deposited film. Photocatalytic tests and contact angle measurements have also been conducted, to confirm the deposition conditions corresponding to the production of the most effective film in terms of photo-reactivity for dye degradation and hydrophilicity.

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# Electrical insulation performance of aluminum oxide layers on metallic substrates – HiPIMS compared to RF-MS

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The utilization of electrically insulating layers like silicon oxide or aluminum oxide is state of the art in the semiconductor industry. Generally, silicon wafers are used because of their excellent characteristics in terms of polishing, structuring, doping, oxidization as well as the application in large-scale production. In comparison, the insulation of technical surfaces with thin oxide layers as it is the case for metallic substrates is more challenging. Yet, these layers are needed for example for thin film sensors to be applied directly onto an arbitrary workpiece.

In this evaluation aluminum oxide layers with thicknesses in the range of 300 nm to 4000 nm are investigated for their electrical insulation capability by measuring the resistance between the substrate and a subsequent conductive layer. The deposition was done in a non-reactive RF-magnetron sputtering process and in a reactive HiPIMS process, respectively. The substrate materials tested are the steel 1.3505 (100Cr6), the titanium alloy 3.7164 (Ti6Al4V) and the aluminum alloy 3.1645 (AlCuMgPb) with both, turned and polished surfaces.

Besides resistance measurements, the layers were investigated by SEM and characterized by EDX. During the HiPIMS deposition the value of the electrical current over the time of one pulse was measured additionally.

The comparison of the resistance values showed strong dependencies between the electrical insulation and the substrate materials used, as well as their surface microstructure. Moreover, a deviating insulation performance for the layers deposited by HiPIMS was proven.  From the results, a correlation of substrate material, surface microstructure and the corresponding deposition technology is deduced.

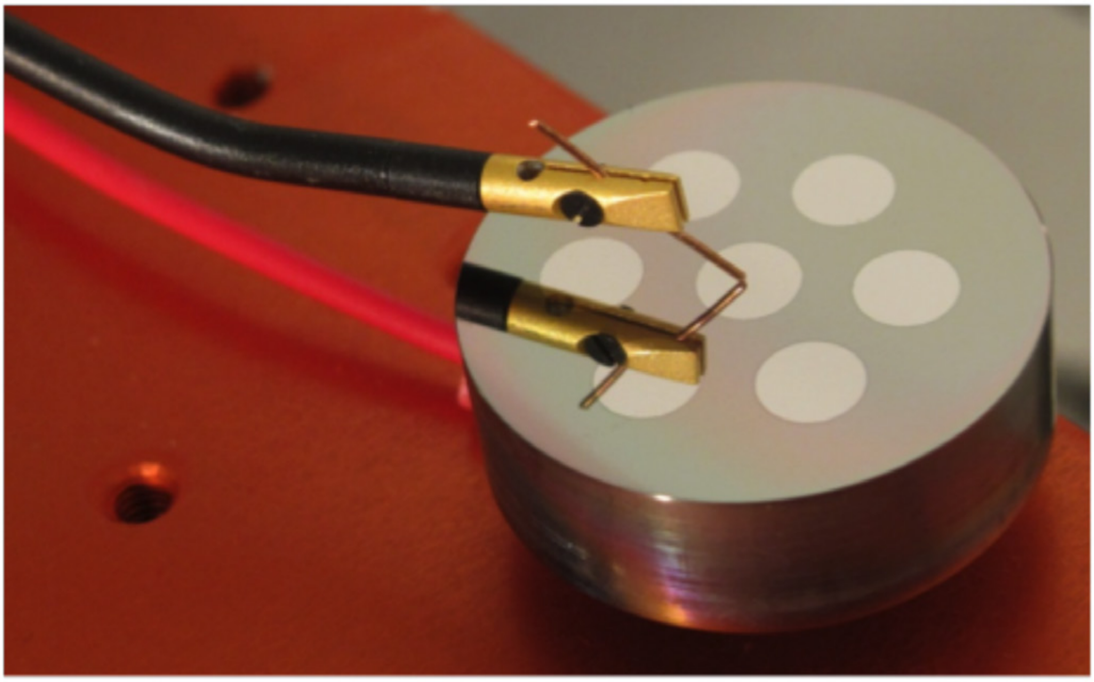

**Figure 1:**

Metallic sample insulated by an aluminum oxide layer during resistance measurements

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# Target and substrate composition in reactive high-power impulse magnetron sputtering — a modelling study

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Reactive high-power impulse magnetron sputtering (HiPIMS) has recently been used for preparation of various optically transparent non-conductive metal oxides, such as $TiO_2$, $ZrO_2$, $Ta_2O_5$, $HfO_2$, optically transparent conductive oxides, such as InSnO, Al-doped ZnO, or thermochromic $VO_2$ films [1]. Improved film properties compared to films prepared by dc magnetron sputtering were reported. The relations between the internal processes in the reactive HiPIMS discharge and the measured discharge characteristics (such as current waveforms) are not fully understood. We have published a parametric model of the reactive HiPIMS process [2] which can give some insight into how the internal discharge processes influence the composition of the target and substrate. The model is fully time-dependent and it takes into account specific features of the HiPIMS discharges, namely gas rarefaction in front of the sputtered target, backward flux of the ionized sputtered metal atoms and reactive gas atoms onto the target, and high degree of dissociation of reactive gas molecules in the flux onto the target and substrate.

In this contribution, we present time evolutions of processes on the target, such as chemisorption and implantation, during HiPIMS depositions of $ZrO_2$ calculated for various discharge conditions corresponding to our experimental work. We focus on the quantification of contributions of these processes to the stabilized compositions of the target and the substrate. We explain the effects of the new model features, especially the dissociation of the reactive gas in the plasma and the return of ionized sputtered metal atoms onto the target. The model calculations indicate that these processes are critical for obtaining stoichiometric films at high deposition rates by HiPIMS.

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# Deposition of titanium oxide films by current-controlled high power impulse magnetron sputtering

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High density transparent oxide layers on glass can improve the environmental protection ability of low emissivity layers in glazing and the photocatalytic deactivation of organic contaminants. High Power Impulse Magnetron Sputtering (HIPIMS) produces high density microstructures due to the delivery of a highly ionised deposition flux to the substrates. The presence of oxygen in the sputtering plasma results in the production of negative ions with an energy close to the sputter voltage which crystallise the coating but may induce lattice defects and stress. Due to the low duty cycle of HIPIMS power pulses, the ions are produced for a short fraction of a pulse period compared to conventional pulsed or continuous sputtering.

TiOx films were produced in a cluster tool by reactive HIPIMS of a pair of metallic targets in an Ar-Oxygen atmosphere. The HIPIMS process was carried out by maintaining a constant current within the pulse. This resulted in the elimination of stability issues associated with runaway currents and target poisoning for oxygen flows ranging from 10 to 50% of the total gas flow. The films were deposited without additional heating or substrate biasing and had good transparency. The thickness uniformity was < 2% across a 100x100 mm area. The refractive index increased continuously as the oxygen flow reduced from 45 to 13% reaching a maximum value of 2.55 at a wavelength of 550 nm. The films were metallic (non-transparent) at 10% Oxygen flow. The films comprised a mixture of rutile and anatase phase with HIPIMS deposition producing higher fractions of rutile compared to bipolar pulsed DC operation. The HIPIMS films reached higher refractive index of 2.55 compared to 2.47 for bipolar pulsed DC. The hardness of the films and its relation to process conditions are discussed.

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# Wave phenomena and instabilities in direct current magnetron sputtering and high power impulse magnetron sputtering plasmas

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Magnetron plasmas are prone to a wide range of wave phenomena and instabilities. Their impact on particle transport and efficiency, especially for high power impulse magnetron sputtering (HiPIMS), is still under discussion. Another interesting question is the transition from a seemingly homogeneous plasma torus for direct current magnetron or low power pulsed plasmas towards the spoke regime in HiPIMS with very distinct and localized travelling ionization zones. Therefore, experiments were conducted using a 2-inch magnetron with a chromium target and argon as plasma forming gas, covering the full range from direct current magnetron sputtering (dcMS) to HiPIMS.

Fluctuations in the floating potential were measured with an array of twelve flat electrical probes. These were equally distributed in azimuthal direction and separated by 30. The radial distance to the racetrack was 17 mm and the distance to the target 5 mm. All pins were simultaneously measured with a 14-bit DAQ. Empirical mode decomposition (EMD) was used to extract the fluctuations and hence to detrend the obtained data. Here, EMD acts as a dyadic

filter bank to decompose nonlinear and nonstationary time series into a finite set of so-called intrinsic mode functions (IMF). Amplitude and frequency modulations described by the IMFs are then clearly separated. From these pre-processed time series it was possible to estimate frequency and wave number spectra.

A two fluid model for non-magnetized ions, magnetized electrons including ionisation and neutral collisions was formulated. The equations of motion and continuity were linearised and the whole analysis was performed in Fourier space. In following this standard procedure, a dispersion relation was derived which was used to fit the experimental data. The obtained results will be presented for dcMS and HiPIMS plasmas and implications on the current understanding will be discussed.

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# Observation of breathing modes in high power impulse magnetron sputtering plasmas

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Investigations of the well-known traveling ionization zones, also known as spokes, by a set of probes revealed evidence for breathing modes: periodic variations of plasma parameters with characteristic frequencies between 10-100 kHz. A set of probes, azimuthally distributed along the racetrack, showed synchronous oscillations of the floating potential at all probes. They appear most clearly in a current regime high for direct current magnetron sputtering but low for the HiPIMS mode. Breathing oscillations were found to be superimposed on azimuthal spoke motion. Depending on pressure and current, one can also find regimes of more-or-less chaotic fluctuations, and stable discharges at high current. A survey of a range of parameters expanded to low current dc discharges provided the opportunity to arrive at a preliminary pressure-current phase diagram showing spoke and breathing modes as well as the stable discharge regime. Layers are essential, critical features for the operation of the discharge and properties of particle fluxes.

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# Anomalous cross-B field transport and spokes in HiPIMS plasma

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The rotation of localised ionisation zones, i.e. spokes, in magnetron discharge is investigated as a function of discharge current, ranging from 10 mA (current density 0.5 mA $cm^{-2}$) to 140 A (7 A $cm^{-2}$). The presence of spokes throughout the complete discharge current range indicates that the spokes are an intrinsic property of a magnetron sputtering plasma discharge. Up to discharge currents of several amperes (the exact value depends on the target material), the spokes rotate in a retrograde E×B direction with angular velocity in the range of 0.2–4 km/s. Beyond a discharge current of several amperes, the spokes rotate in a E×B direction with angular velocity in the range of 5–15 km/s. In this paper we present experimental evidence that anomalous diffusion is triggered by the appearance of spokes rotating in the E×B direction. The Hall parameter $\omega_{ce}T_c$, product of the electron cyclotron frequency and the classical collision time, reduces from Bohm diffusion values (~ 16 and higher) down to the value of 3 as spokes appear, indicating anomalous cross-B field transport. The ion diffusion coefficients calculated from a sideways image of the spoke is six times higher than Bohm diffusion coefficients, which is consistent with the reduction of the Hall parameter.

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# HiPIMS plasma measurements by triple and target strip probes

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The triple probe is a useful diagnostic tool for measuring the plasma electron density $N_e$ and electron temperature $T_e$ in low-pressure plasma systems without the necessity to collect a current-voltage probe characteristic. Measurements can be made with time-resolutions down to a few ns, making the acquisition of plasma parameters possible in rapid single shot events, inaccessible to Langmuir probes. In this study, we us a triple probe to measure $N_e$, $T_e$, the floating $V_f$ and plasma potential $V_p$ associated with rapid azimuthally rotating ionization zones (known as spokes) observed above a niobium target in HiPIMS discharge operating in argon gas. The triple probe was used in conjunction with a strip probe embedded in the target which provided point information on the transient discharge ion current $I_D$ due to the spokes.

Triple probe measurements taken 15 mm above the target surface in the magnetic trap of the racetrack region revealed an amplitude of modulation in $N_e$ and $T_e$ of approximately 50% compared to their base values. Perturbations in ID were found to be closely correlated with those in the spoke parameters and to be about 75% of the base discharge current value under the same conditions. These modulations have frequencies of approximately 170 kHz, in line with typical values reported. Phase information showed that in the spoke, the peak in $N_e$ leads that of $I_D$, closely followed in time by the peak in Te, preceding a trough in these parameters. The troughs for ID and Ne were completely in phase. The phase of $V_f$ and $N_e$ are very similar as are the phase of $V_p$ and $I_D$. The phase difference between $T_e$ and $I_D$ can be explained by the release of secondary electrons from the target, causing local plasma heating; this is supported by the correlation between $I_D$ and $V_p$ correlation. The delay between the peaks of $N_e$ and $I_D$ is unexpected, but is understandable given the gap between the target and triple probe.

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# Optimized magnetic field configuration for high power impulse magnetron sputtering

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High quality thin films with enhanced density, adhesion, structure, etc, can be obtained with High Power Impulse Magnetron Sputtering (HiPIMS). Although the film quality is superior in comparison with direct current magnetron sputtering (dcMS), HiPIMS technique did not get much attention from industry due to its lower deposition rates compared to dcMS. The observed lower deposition rates are due to the metal ion “return effect”[1]. The “ε” magnet pack[2] that was developed at the Center for Plasma Materials Interactions (CPMI) at the University of Illinois demonstrated increased deposition rates in HiPIMS compared to the conventional magnet pack arrangement. However, the magnetic fields in the “ε” magnet pack are highly asymmetric. Therefore, an upgraded cylindrically symmetric TriPack magnet pack with increased deposition rates in HiPIMS was developed to create better substrate uniformity. The most recent results from this new TriPack magnetic field design are discussed. The topics include: an investigation of the moving localized “ionization zones” in the TriPack with a gated ICCD camera, measurements of the ion fraction of sputtered material, and the deposition rates from various target materials. The films deposited with the Starfire Impulse 2kW HiPIMS dual power supply were compared with films grown with conventional methods. The Starfire Impulse power supply used in this work has an option to apply bias on the substrate that can be synchronized or delayed to the HiPIMS voltage pulse for superior film properties.

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# Optical emission spectroscopy of a controlled reactive HiPIMS during a high-rate deposition of densified stoichiometric ZrO2 films

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Effective deposition of dielectric oxide 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 in order to utilize exclusive benefits of the HiPIMS in a high-rate reactive deposition of oxide films. Depositions were performed in a stainless steel vacuum chamber (diameter of 507mm and length of 520mm) equipped with an unbalanced magnetron (Zr target, the diameter of 100mm). Oxygen was admitted into a vicinity of the magnetron target via two corundum conduits with the inlets (diameter of 1mm) directed to the target (to-T case) or to the substrate (to-S case). 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, $I_d$ , that oscillates in compliance with the instantaneous local $O_2$ partial pressure in the discharge.

Here, we report on the results of the optical emission spectroscopy with a temporal and a spatial resolution carried out during the deposition of densified stoichiometric $ZrO_2$ films using the HiPIMS controlled by the pulsed RGFC system. Light from the discharge was collected by a collimator and analyzed by a monochromator equipped with a photomultiplier connected to a photon counter. Counts of photons for the selected emission lines were recorded with the temporal resolution of 320ns around the minima and maxima of $I_d$ The influence of the O2 inlet direction and of the preset depositionaveraged target power density $<s_d>$ was examined for voltage pulses with the duration of 200μs and the repetition frequency of 500Hz at the Ar partial pressure of 2Pa.

We have found that the almost ten times rise in $<s_d>$ (from 5.4Wcm$^{-2}$ to 51–52Wcm$^{-2}$) leads to a radical increase in the sputtering rate of the target atoms and in the ionization rate of plasma species. This results in large ion fluxes of the sputtered target material and reactive gas particles to the substrate. Moreover, a quick rise in the Zr atom populations at voltage pulse beginnings for high-power regimes is an evidence that the target surface is only slightly covered by a compound. From the population of $Zr^+$ ions, the time evolutions of the excitation temperature, that follows trends of the electron temperature, were determined. For the high-power cases, the values of after an initial peak remain almost constant from approximately 20μs after the voltage pulse initiation up to its end and they are systematically lower for higher values of the local $O_2$ partial pressure in the discharge. Since the ratios $n_m/J_t$ where $n_m$ is the population of a radiative state and is the instantaneous target current density, reflect trends of species ground state densities near the target when ≈ const., the time evolution of these ratios for the monitored emission lines were constructed (see the constancy of $T_{ex}$ to reveal a behavior of the corresponding species ground state densities during a voltage pulse. The $n_m$ ratios have shown that the Ar atom density is strongly reduced near the target at maxima for the high-power regimes due to their ionization and a very intense sputtering wind of Zr atoms. The density reduction is also evident in the ground state densities of O atoms for both inlet directions, but for the to-S case the reduction is not apparent in $O^+$ ions density. This may indicate that the reduction in O atoms density is caused mainly by their ionization. Moreover, in spite of the $O_2$ flow rate oscillations on scale of seconds adjusted by the pulsed RGFC system during the deposition, we observe only small differences in the densities of Zr, Ar and O atoms and ions corresponding to the highest and lowest local $O_2$ partial pressure in the discharge for the optimized to-S case. This shows that the pulsed RGFC system keeps the HiPIMS deposition process close an optimal point for the high-rate deposition of densified stoichiometric $ZrO_2$ films (the deposition rate up to 120nm/min was achieved on a floating substrate in the distance of 100mm from the target).

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# Plasma analysis of inductively coupled impulse sputtering by investigation of Cu, Ti and Ni species

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Highly ionised pulsed plasma processes have helped to improve coating properties by enabling the control of flux of sputtered ion species.

Deposition of magnetic materials, such as Nickel, is problematic with magnetron sputtering as the magnetic field necessary for the sputter process is reduced due to quenching of the magnetic field by the target material.

With Inductively Coupled Impulse Sputtering (ICIS) we can remove the magnetron. To generate the plasma, pulsed RF power is applied to an internal coil. Ar ions are attracted to the target surface by utilising high power DC pulses on the cathode and initiate sputtering. The sputtered material is then ionised as it passes through the coil volume, creating a highly ionised metal flux to the substrate.

For ICIS the creation rate for gas, metal and metal ion species are unknown.

In the conducted experiments both DC and RF power supplies were running at a duty cycle of 7.5% and were synchronised to ensure the pulse on time was the overlapping. A power-pressure matrix was created to examine the influence on the plasma. At a constant pressure of 13 Pa the applied RF power was varied from 1000-4500 W, at constant applied RF power of 3000 W the pressure was varied from 3-26 Pa. The pulsed DC voltage to the target was kept constant at 1900 V. OES, energy resolved MS and I-V curve measurements were taken for each examined element.

OES measurements for increasing power have shown a linear increase in intensity with increasing power. The slopes of gas and metal species in a log-log graph exhibit a factor 1 increase from the Ar neutral intensity for each excitation and ionisation step, suggesting electron collisions to be the main excitation mechanism.

The IEDFs measured by MS show two sharp peaks, one high intensity peak at 20 eV which corresponds with the plasma potential and is ideal for increased surface mobility without inducing lattice defects. The second lower intensity peak, of high energetic ions, is visible at 170 eV.

We will be discussing the voltage and current waveforms relationships of the slope factor ß in an intensity-power graph, the IEDFs vs. power and pressure relations and the origin of higher energetic ions.

# Space-resolved plasma diagnostics in a dcMS/HPPMS hybrid (Cr,Al)N process

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## Abstract

The high power pulse/impulse magnetron sputtering (HPPMS/HiPIMS) has been frequently studied in the last years. Coatings deposited by means of HPPMS exhibit lower roughness, denser microstructure and better mechanical properties in comparison to coatings deposited by direct current magnetron sputtering (dcMS). This is caused by the low pulse duration ton and the resulting high peak power $P_p$ at the cathode and ionization of the plasma. However, the low duty cycle causes a decreasing deposition rate of HPPMS compared to dcMS. From an economic point of view this is a disadvantage of the HPPMS technology. Therefore, the dcMS/HPPMS hybrid technology has been newly introduced to overcome this issue. Within the dcMS/HPPMS hybrid process dcMS and HPPMS cathodes are used simultaneously to combine the benefits of both technologies. It is known that the coating properties of a dcMS/HPPMS hybrid coating can be arranged between pure dcMS and HPPMS processes. Nevertheless, there have been no detailed investigations on the influence of different dcMS/HPPMS plasma zones on the coating properties. Therefore, in the present work measurements on plasma properties as well as on coating properties using a dcMS/HPPMS hybrid (Cr,Al)N process were carried out in an industrial scale PVD coating unit. Two Cr targets with 20 plugs of Al ($CrAl_{20}$) were used. One target was mounted on the HPPMS cathode and the other on the dcMS cathode. The dcMS cathode was powered with $P_{dcMS}$ = 3 kW. The HPPMS cathode was powered with $P_{HPPMS}$ = 5 kW. The HPPMS pulse length was $t_{on}$ = 40 µs and the pulse frequency f = 500 Hz. The plasma was analyzed space-resolved to map the entire area in front and beside of the cathodes. Changes in the plasma composition were investigated by optical emission spectroscopy (OES). The time-resolved ion energy distribution function (IEDF) and the ion current density were investigated by retarding field energy analyzer (RFEA). Furthermore, (Cr,Al)N coatings were deposited on samples with fixed positions within the coating unit, which were comparable to the space-resolved plasma diagnostics. The microstructure of the coatings was investigated by means of scanning electron microscopy (SEM). The universal hardness and the indentation modulus were determined by nanoindentation. Furthermore, the chemical composition of the coatings was analyzed by energy dispersive X-ray spectroscopy (EDX). The space-resolved influence of the dcMS/HPPMS hybrid process on the plasma and coating properties was deviated from correlations of the measurement data.

**Keywords:** PVD, dcMS/HPPMS, HiPIMS, (Cr,Al)N, RFEA, OES

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# The effect of Cr content on microstructure and mechanical properties of AlCrTiN films deposited by a hybrid system with HIPIMS and dcMS

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## Abstract

The AlTiCrN coatings with different Cr contents were deposited from AlTi and Cr targets using a hybrid coating process combining high power impulse magnetron sputtering (HIPIMS) and a DC magnetron sputtering (DCMS). The HIPIMS mode was applied to the AlTi target with a fixed average power of 10 KW while the Cr target was operated in DCMS mode with varying power from 0 to 10 KW. The deposition temperature and bias voltage was fixed at 450 °C and -100 V, respectively. The microstructure and mechanical properties of the AlTiCrN coatings were investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), and nano-indentation. The tribological behavior of the AlTiCrN coatings were evaluated by pin-on-disk tribo-tests at 400 °C, 600 °C and 800 °C. The results indicated that all the AlTiCrN coatings exhibited (Al,Ti,Cr)N solid solution phase with FCC NaCl-type structure. With increase of Cr target power, the Cr content increased from 0 to 25 at. % tailoring changes in coating microstructure and properties. Firstly, the preferred orientation shifted from (111) to (200). Secondly, the hardness and elastic modulus of the AlTiCrN coatings increased from 31.4 GPa and 448.8 GPa at 0 at.% Cr to 33.7 GPa and 469.5 GPa at 8.9 at.% Cr, and then decreased with further increase of Cr content. Thirdly, the friction coefficient and wear rates of the coatings against $Al_2O_3$ balls also exhibited some relationship with the coating compositions and test temperatures. The inherent mechanism was analyzed and discussed.

**Keywords:** AlTiCrN coatings; hybrid coating process; high power impulse magnetron sputtering; coating microstructure ; tribological behavior

# High rate HiPIMS for cutting tool coatings

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A deposition rate as high as possible is a key requirement to every commercial coating process. This paper will introduce a new HiPIMS concept for increasing the deposition rate. The concept is based on the CemeCon door-assembly design, which avoids any cable between pulse unit and cathode, and features a full synchronization between the HiPIMS sources and a dedicated table Bias. Plasma characterization demonstrates that this results in highest ionization. Together with reduced re-sputtering this novel process regime gives a so far unachieved deposition rate for HiPIMS. Case studies show how this new hardware and process design turns the advantage of the HiPIMS technology such as enhanced film adhesion, denser morphology and better coating uniformity all around 3D objects into user benefits for cutting tool applications.

A lot of research is currently dedicated to the machining process of titanium and heat resistant super alloys based on nickel, iron or cobalt. Jet engines and gas turbines made of this material group operate at a higher working temperature and thereby raise the energy conversion efficiency. Key obstacles to productive metal processing are the extreme cutting temperature, the high strength and the tendency to stick to the carbide substrate of the tool. $TiB_2$ films are a promising candidate due the high hardness of this ceramic material and its low affinity to non-ferrous metals. This next step in the development of HiPIMS broadens the application range of $TiB_2$ from the tradition aluminium manufacturing sector to super alloys.

Case studies show how a dedicated HiPIMS process leads to fine-grain $TiB_2$ morphology. The film shows hardness levels above 4.000HV – which is typical for $TiB_2$ films – combined with low young's modulus. High toughness makes it rather suitable for operations like thin wall milling for jet engines. Milling tests in the aircraft sector demonstrate how the superb adhesion of HiPIMS supports the machining of titanium and super alloys further.

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# Performance comparison of DC and HiPIMS TiAlN coatings in metal cutting

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The manufacturing industry is constantly trying to improve machining processes to reduce cost and lead time. It has turned out that the use of advanced cutting tools and especially hard coatings is decisive. The introduction of HiPIMS technology for tool coating has enabled a further step in productivity. In this study, we compare the milling performance of commercial DCMS and HiPIMS TiAlN coatings on milling inserts. Inserts from the same production lot were used coating so that any difference in tool performance must reflect differences between the coatings. The tests were carried out in alloyed steel DIN 42CrMo4 and ductile cast iron EN GJS-600. The cutting speed and carbide substrate was varied. We performed a thorough characterisation of the coatings with regards to their chemical and mechanical properties. This included SEM, EDX, and XRD. Overall, the HiPIMS coated inserts showed longer tool-life. We could attribute the differences in wear behaviour to coating properties such as more favorable H/E ratio. We found that especially crater wear could be reduced by the HIPIMS coating variant (Fig. 1). One of the reasons for this seems to be improved adherence of pulse sputtered coatings. Interestingly, by using a HiPIMS coating, less wear resistant substrates intended for cutting of steel could also be used for harder materials like cast iron with improved results. This underlines the huge potential of advanced coating technology for optimisation of manufacturing processes.

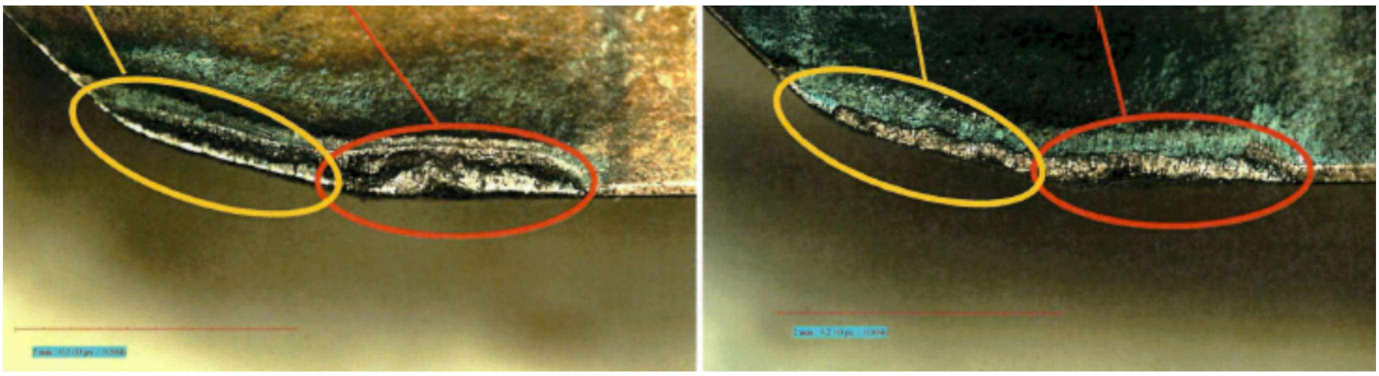

**Fig. 1:**

Crater wear on a carbide milling insert used on GJS-600 cast iron at similar cutting length, (a) insert coated with DCMS TiAlN, (b) insert coated with HiPIMS TiAlN.

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# Tribological behaviour of Mo–W doped carbon-based coating

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## 1. Introduction

Metal-free and metal-doped diamond-like-carbon (DLC) coatings are extensively used as tribological coatings for engine parts due to their excellent combination of low friction and improved wear resistance properties. Engine components such as piston rings, piston pins, cam followers, cam shafts, rockers, gears and tappets are often coated with DLC and involve interfacial contacts with either steel or DLC coated surfaces in the presence of lubricants.

But the automotive industry is changing rapidly and the requirements for components are altering as well. Due to downsizing of the engines, operating conditions are becoming ever harsher, which means the components and the applied coatings have to withstand much higher temperatures and mechanical loads.

## 2. Mo-W Carbon based coating

A novel carbon-based coating doped with molybdenum and tungsten (Mo–W–C) has been developed using the advantages of HIPIMS technology. Molybdenum and tungsten are doping elements producing "in situ" lubricious phases such as $MoS_2$ and $WS_2$ via tribochemical reactions with the oil lubricant. This Mo-W-C coating was developed in order to provide low friction in boundary lubricated sliding condition at ambient and at high temperature. Additionally, both elements enhance high temperature stability of the coatings.

## 3. Results

The Mo–W–C coating showed the lowest friction coefficient among a number of commercially available state-of-the-art DLC coatings at ambient temperature. At elevated temperature (200°C), Mo–W–C coating showed a significant reduction in friction coefficient with sliding distance in contrast to DLC coatings. (Fig. 1)

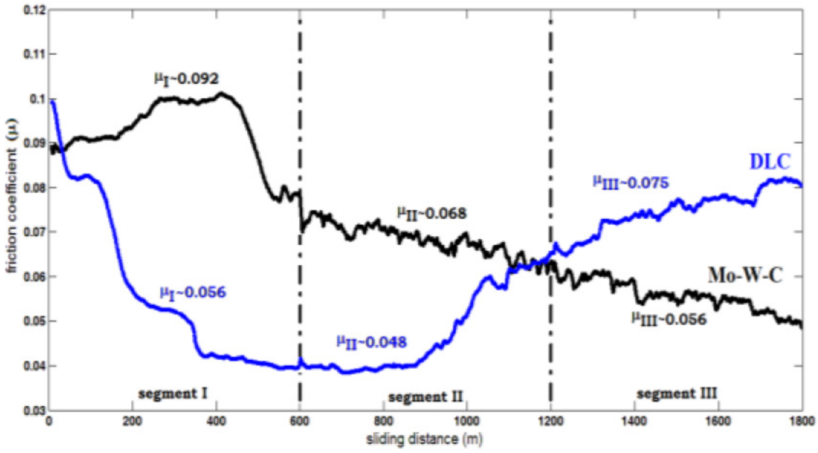


**Fig. 1:**

COF of Mo-W-C and DLC films in lubricated sliding at 200°C against a 100Cr6 counterpart

The molybdenum and tungsten dopants contribute to an improved high temperature stability of the carbon based coating. In thermo-gravimetric experiments, DLC coated samples begin to lose mass due to oxidation and spallation at temperatures as low as 300°C, whereas the Mo-W-C film retains its mass unchanged up to 600°C in the same experiment.

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# Advanced power delivery control in High Power Impulse Plasma Magnetron Sputtering process

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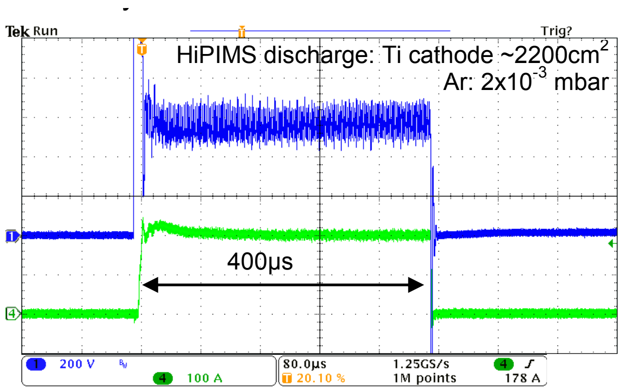
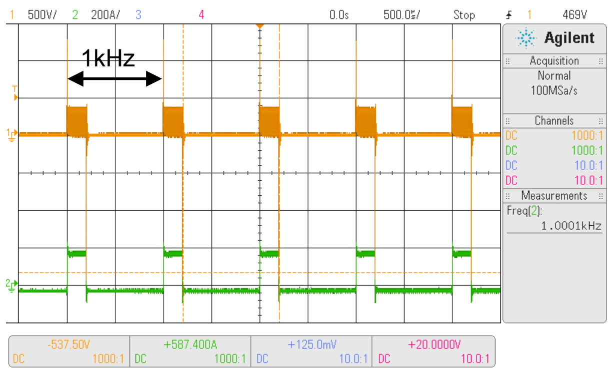
Since the first presentation of the High Power Impulse Magnetron Sputtering (HiPIMS) idea by Kouznetsov and co-workers in 1999 the basic architecture of a DC-charged capacitor bank dissipating periodically its energy into the plasma in pulses evolved to a sophisticated electronic device commercially available on market for industrial application.

In order to meet rigorous requirements of academia and industry, engineers have proposed different modifications of HiPIMS power delivery units to make the pulse shape and duration independent on the size of the capacitor bank and time-dependent plasma impedance. Since typically pulses longer than 100 µs are required to reach metal self-sputtering regime, a precise control of voltage and current pulse shape is required during the whole pulse length. Furthermore, for tuning ionization of the plasma species and deposition rate flexibility in power delivery regulation is of a key importance.

In this contribution a novel approach of a flexible and application selectable HiPIMS power delivery control of the TruPlasma Highpulse Series will be described. Preliminary in-field experiments will be used to depict introduced algorithms. First the peak current regulation mode and its implications on the ionization degrees of the sputtered species will be analyzed.




As next, the average power regulation mode which includes pulse length and frequency modulation will be introduced. The discussion will be completed with a summary of an automatic pulse length control activated to compensate deposition losses in a case of arc suppression event.

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# HiPIMS Power Supply Technology

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In spite of the great perspective and the most positive outlook forecasts given by scientists and technologist in the past decade for the HiPIMS-technology, the real industrial breakthrough has not yet started. HiPIMS is still considered as a niche technology. Partly this could be the case because the HiPIMS power supply technology up to now was not yet readily industrialized. HiPIMS-PS were very expensive, not very reliable and in some cases even unsafe to use. The Deposition rate of HiPIMS vs conventional sputtering is still considered to be rather low.

Taking all this into account an experienced team of scientists, researchers, technologists, metallurgists, process engineers and last but not least a highly skilled power supply manufacturer got together with the goal to contribute to the industrialization of the HiPIMS-technology with a sophisticated HiPIMS-PS. The hiP-V collaboration was found.

Designed as a multi-purpose “All in One” system, the hiP-V HiPIMS-PS can be used for multiple applications as a HiPIMS-PS, a HiPIMS Bias-PS and as a DC/DC-pulse PS. Uni-Polar or Bi-Polar processing as well as superimposed or sequential HiPIMS (in combination with DC or MF) is possible. All this, for single or dual magnetron applications.

Unique for HiPIMS power supplies is the high modularity and flexibility of the hiP-V products. The hiP-V HiPIMS-PS can be connected either in serial or in parallel to increase the voltage or the current / power which is to be applied on the magnetron. This, the wide power range and the fast ARC-handling make the hiP-V product line dedicated for high volume industrial production at reasonable costs.

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# Influence of high voltage pulsed bias on surfaces treated by HiPIMS

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As is well known, HiPIMS processing allows coating deposition on complex structures ensuring homogeneous layer thickness [1]. Innovative combination of HiPIMS and plasma based ion implantation (PBII) offers new possibilities for homogeneous surface modification taking advantage of the high density of target ions and high ionization rate. This technology enables coating and doping of surfaces successively or even simultaneously.

We present recent results on measuring the energy flux onto substrates pulsed with high voltages up to several kV during HiPIMS based Cu deposition. The measurements have been performed by a calorimetric probe [2] analyzing the time dependent change in probe temperature. Due to limitations in probe design it is specifically challenging to determine the energy flux onto high voltage pulsed substrates. Therefore, a biased grid was used between HiPIMS source and calorimetric probe. Influences of delay time and distance between substrate and magnetron onto the energy flux for a high voltage pulsed substrate have been investigated. The results exhibit the remarkable effect of delay time between HiPIMS and PBII pulse inter alia revealing a parameter setup by which a maximum ion and energy flux towards the substrate can be obtained.

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# Low-temperature growth of dense, hard $Ti_{1-x-y}Al_xTa_yN$ alloy films via hybrid HIPIMS/magnetron co-sputtering using synchronized metal-ion irradiation

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## Abstract

Hard $Ti_{1-x}Al_xN$ thin films are of great importance for industrial cutting applications. The hardness, thermal stability, and oxidation resistance of these coatings can be further enhanced by alloying with TaN.[1]

Here, we use a hybrid high-power pulsed and dc magnetron co-sputtering (HIPIMS/DCMS) technique[2,3] to grow dense and hard $Ti_{0.41}Al_{0.51}Ta_{0.08}N$ alloys without external heating (Ts < 160 °C) in an industrial magnetron sputtering system. While separate Ti and Al targets operating in DCMS mode maintain a high deposition rate of ~50 nm/min, the heavy $Ta^+/Ta^{2+}$ irradiation from the HIPIMS-powered Ta target synchronized with a pulsed dc bias provide effective near-surface atomic mixing resulting in film densification. Between the short bias pulses (2% HIPIMS duty cycle) the substrate is at floating potential to minimize $Ar^+$ bombardment that typically leads to high compressive stress.

Scanning electron microscopy (SEM), fig. 1, reveals dramatic differences in the microstructure of the films grown at floating potential with all targets in DCMS mode (Ta-DCMS) compared to the co-sputtered HIPIMS/DCMS (Ta-HIPIMS) films with the same composition. While the Ta-DCMS films exhibit both inter- and intra-columnar porosity resulting in 73% density (relative to 5.20 g/cm$^3$ for a fully-dense alloy film with 8 mol% TaN and $a_o$ = 4.24 Å), the Ta-HIPIMS layers possess narrow column boundaries, essentially no inter-columnar porosity, and a relative density of 93%. The Ta-HIPIMS films also exhibit smaller grain size and lower surface roughness than the Ta-DCMS layers. The mechanical properties are significantly improved with an increase in hardness and elastic moduli from 15.3 and 289 GPa for reference Ta-DCMS films to 28.0 and 328 GPa for $Ti_{0.41}A_{l0.51}Ta_{0.08}N$ layers grown by HIPIMS/DCMS co-sputtering.

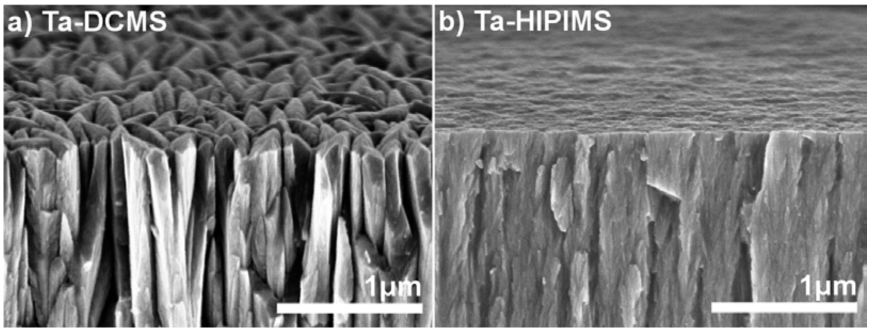


**Figure 1:**

SEM images of $Ti_{0.41}Al_{0.51}Ta_{0.08}N$ thin film grown by a) pure DCMS b) HIPIMS/DCMS co-sputtering

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# Deposition of Cu/Mo Multilayers by Bias HiPIMS for X-Band accelerating structures

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## Abstract

The next generation of linear accelerators is highly demanding in terms of accelerating gradients. To enhance the performance of X-band Linacs at multi- GHz many resources are devoted to achieve accelerating gradients higher than 100MV/m and to obtain the highest possible electrical breakdown reliability. In the framework of a large collaboration among SLAC (USA), KEK (Japan), UCLA (Los Angeles) and INFN, LNL have been involved in the development of new materials for X-Band cavities. Several bulk materials are used in the development of these cavities but the use of physical vapor deposition to obtain nano-structured engineered surfaces directly on these small sized cavities is not reported. As a matter of facts the inside size of these cavities is of order of ten millimeters and typical beam iris apertures ranges from 2 to 6mm: with these dimensions the direct PVD coating from inside is impossible, therefore the mandrel (Figure 1) representing the negative of the cavity obtained with micrometer precision milling and turning and nanometer polishing is first PVD coated and then chemically dissolved after copper electroforming

Since the shape of the mandrel comprises deep grooves with almost vertical walls and aspect ratios in the range 4÷5, ionized sputtering has been chosen as the PVD deposition technique. With this method, by applying a negative bias voltage to the mandrel during deposition, the metal ions may reach the grooves' walls with defined energy and with impinging angles near to normal producing denser coatings with better wall coverage. The research activity carried out at LNL aims at developing nanostructured metallic coatings suitable to improve the RF breakdown performance of the accelerating structures. The main task is the optimization of deposition parameters and the characterization of novel nanostructured coatings with different materials (homogeneus X-Cu or alternating X/Cu with X=Mo, W, Ta, Nb).

Multilayer coatings are obtained with the new dual Target High Power Impulse Ionized Magnetron Sputtering (HiPIMS) system in closed field unbalanced magnetic configuration designed at LNL. The negative DC Bias is applied to the mandrel through an electrical network that prevents damaging the bias power supply as a consequence of the high pick-up currents. The system is equipped with two HiPIMS power supplies (Hüttinger 4001 and SINEX 1.2). Optical Emission Spectroscopy (OES) is used to study the ionized metals. Ar N60 is used as plasma gas at pressures in the range 4.5÷7.0 $10^{-3}$ mBar. The mandrel is kept in continuous rotation and water cooled during Biased HiPIMS. The target-to-mandrel distance may be varied from 10 cm to 21cm.

The feasibility of depositing Cu/Mo multilayers with single layer thickness and periodicity in the nanometer range has been carried out successfully. EBS and STM results will be presented and correlated to the deposition conditions.

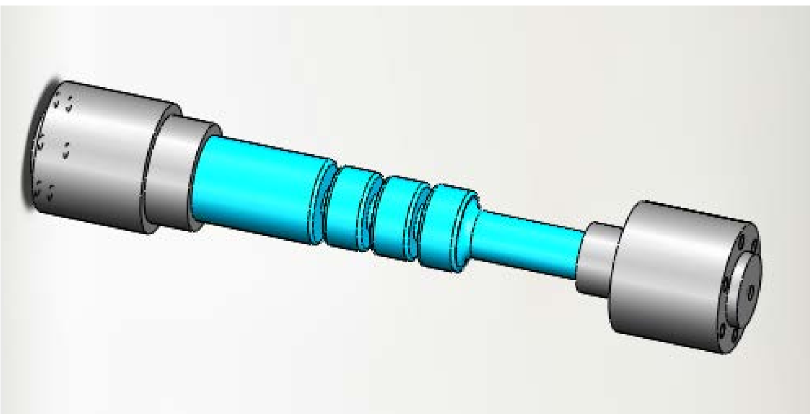

**Figure 1:**

Example of mandrel used to elctro-form a
3 cells X-Band cavity.

# Copper thin films deposited under different power deliver modes and magnetron configurations: A comparative study

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The highly-ionized plasma in high power impulse magnetron sputtering (HiPIMS) discharge opened the possibility of growing denser thin films with less surface roughness and superior hardness and wear properties compared to the conventional sputter technologies. The ionization rate of sputtered species and energetic ion flux towards the substrate can be controlled through the pulsing scheme, as well as through the magnetic field’s strength and configuration.

The aim of this work is to compare the topological, mechanical and tribological (hardness, Young’s modulus, adhesion/cohesion, coefficient of friction) and structural properties of 800 nm nanocrystalline copper (Cu) thin films deposited on silicon substrates by conventional dc magnetron sputtering (dcMS) and HiPIMS operated with single ultra-short pulses (3μs), in the absence and presence, respectively, of an additional magnetic field.

Atomic force microscopy, nanoindentation and nanoscratch measurements were carried out in order to study the surface topography and mechanical and tribological properties of the obtained thin films. Structural properties have been analyzed by X-ray diffraction (XRD). Energy resolved mass spectroscopy was performed in the substrate’s vicinity in order to characterize the ion energy distributions and composition.

It was observed that in HiPIMS, the presence of an additional magnetic field facilitates the transport of charged particles towards the substrate, leading to a higher deposition rate. According to the mass spectroscopy results, the $Cu^+$ ion energy distributions recorded for the HiPIMS discharge assisted by an additional magnetic field are broader with a significantly more energetic tail and contain a larger fraction of highly energetic ions. Information gained from AFM scans revealed that all the samples have uniform surfaces, with RMS roughness values less than 1% of the their thickness. Even so, the HiPIMS-films exhibit significantly smoother surfaces compared with those deposited by dcMS. According to the contact stiffness dependence on the normalized indentation depth, all the film-substrate systems are elastically inhomogeneous, showing soft-hard behaviour. With five indentations, the lowest average values of $H$ and $E$ were found for the dcMS-films, while the hardest coatings (3.1 GPa) with the highest elastic modulus (148.2 GPa) and the highest $H / E$ ratio were found to be the Cu thin films deposited by HiPIMS assisted by an additional magnetic field. The latest ones have also presented the highest amount of elastic recovery and highest adhesion and cohesion. The density of the deposited thin films was obtained by measuring the real atomic density by Rutherford backscattering spectrometry (RBS) and the film thickness by scanning electron microscopy cross-section images.

According to these results, the HiPIMS technique assisted by an additional magnetic field seems to be a good solution for obtaining smooth and dense Cu thin films, with high $H / E$ ratio.

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# Time-resolved ion flux and impedance measurements in reactive high-power impulse magnetron sputtering

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A new planar ion flux probe based on the Sobolewski method for time-resolved plasma characterization in inherently noisy pulsed plasma discharges has been developed. The probe was evaluated in high-power impulse magnetron sputtering (HiPIMS), which is a promising ionized physical vapour deposition technique based on pulsed plasma discharges used to engineer thin films with improved properties. Both non-reactive (pure Ar) and reactive ($Ar/O_2$) deposition processes were investigated using a Ti sputtering target. It was found that the process exhibited a nearly hysteresis-free and stable transition region at the chosen deposition conditions. Time-resolved measurements of the absolute ion flux impinging on the probe placed at the substrate position as well as of the probe sheath impedance during the HiPIMS pulse were recorded in the metal, transition, and compound modes. Gradual changes in the measured ion flux as well as the impedance were seen when transiting from the metal mode to the poisoned mode. It is therefore suggested that this type of robust plasma probe can potentially be used for reactive process control, where the user would like to stably operate in the transition region over long periods of time.

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# Measurement of deposition rate, ionized flux fraction and ion energy distribution in a pulsed dc magnetron sputtering system using a retarding field analyzer with embedded quartz crystal microbalance


## Abstract

A compact retarding field analyzer with embedded quartz crystal microbalance has been developed to measure deposition rate, ionization fraction and ion energy distribution arriving at the substrate location. In the design, the collector plate has been replaced by the front facing electrode of the quartz crystal. The sensor schematics is shown in Figure 1.

The quartz crystal provides a direct measurement of the deposition rate at the substrate location while the analyser grids can be configured to turn on and off the ion flux. In this way the deposition rate can be determined when both neutrals and ions are present and when only neutrals are present. From these two measurements the ionized flux fraction and neutral flux fraction can easily be determined. The sensor can be placed on grounded, electrically floating or radio frequency (rf) biased electrodes and is suitable to characterize HiPIMS and traditional pulsed sputtering processes. A unique calibration method utilising a reference crystal is presented to compensate for temperature effects in the quartz crystal. The metal deposition rate, metal ionization fraction and energy distribution of the ions arriving at the substrate location are investigated in an asymmetric bipolar pulsed dc magnetron sputtering reactor under grounded, floating and rf biased conditions. The diagnostic presented in this research work does not suffer from complications caused by water cooling arrangements to maintain constant temperature and is an attractive technique for characterizing a thin film deposition system.


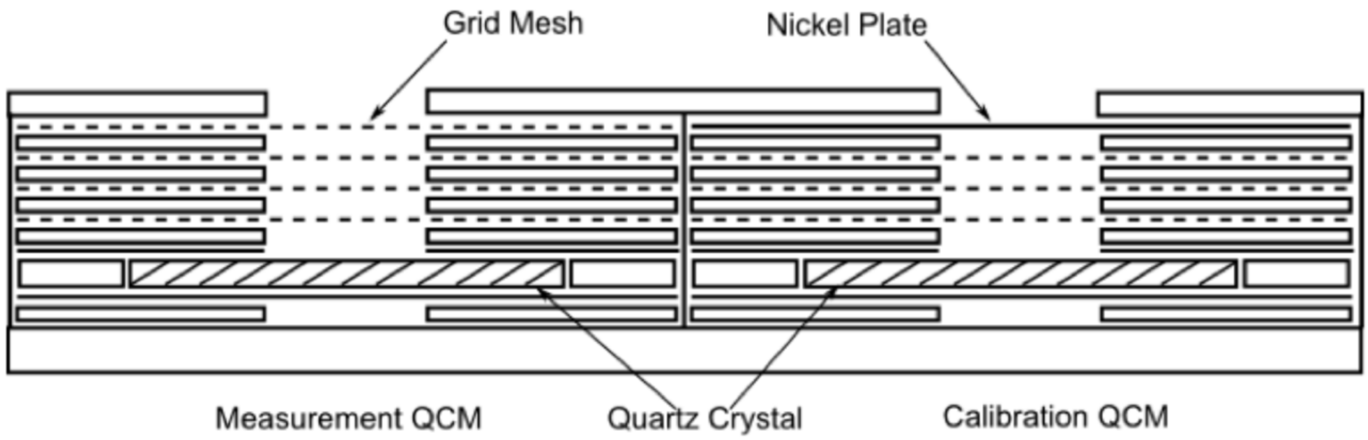


**Figure 1:**

Schematic of the showing the gridded element and the reference crystal


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# Plasma diagnostics in reactive high power magnetron sputtering in Ar+H2S gas mixture

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Semiconducting sulfides as iron pyrite $FeS_2$ and $WS_2$ are recently attractive materials for various photonic applications. Due to its relatively large optical absorption coefficient in the visible region this material can be suitable for applications in photovoltaics, photodetectors and photoelectrochemistry. Semiconducting polycrystalline and nanocrystalline $FeS_2$ and $WS_2$ thin films can be deposited by a high power impulse magnetron reactive sputtering system (R-HIPIMS) by use of metallic target made of Fe or W and in reactive gas mixture of Ar+$H_2S$. The HIPIMS magnetron reactive plasma was investigated during the deposition of this type of sulfide thin films by an optical emission spectroscopy, time resolved Langmuir probe system and by monitoring of heating flux by a calorimetric probe. Furthermore, a synchronized pulsed modulated high frequency voltage with the frequency $f$=250 kHz was applied on the substrate in order to generate controlled ion substrate bombardment at the defined part of HIPIMS pulse. The ion flux on the substrate was determined by a Sobolewski method from RF current and voltage waveforms measured on the substrate. The partial pressure of $H_2S$ in the plasma reactor was changed in a wide range for these experiments. Sulfide thin films were deposited at different magnetron pulsing frequency, magnitude of impulse power and $H_2S$ partial pressure. Some optimum deposition conditions were found. In order to be able to make photoelectrochemical measurements, sulfide films were also grown on Si substrate with Pt electrode and on glass with ITO electrode and $TiO_2$ anatase n-type semiconductor.

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# Properties and process control of reactively sputtered alumina coatings with a novel HIPIMS approach

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Aluminum Oxide ($Al_2O_3$) is a polymorphic material whose properties depend strongly on deposition conditions during sputtering. Substrate temperature has a strong influence on crystal structure. Below temperatures of 450°C, $Al_2O_3$ is typically amorphous. From 450°C – 650°C, gamma $Al_2O_3$ and other mixed phases can be formed. It is possible to deposit alpha $Al_2O_3$ at temperatures above 650°C with standard deposition techniques. The energy, type and flux density of ions impinging on the growing film during the deposition are the other main factors. In standard DC sputtering, mainly neutrals are deposited and gas ions are available for film densification. These species are rather "inefficient" at transferring energy directly to the growing film. With a HIPIMS discharge, the density and energy of Al and O ions can be increased. As the adatom mobility and surface chemistry will be strongly influenced by the presence of such ions, it may be possible to reduce the deposition temperature while maintaining the desired crystal phase. The addition of a positive high voltage pulse directly after the negative high power pulse can also have a significant effect on the coating properties.

In this work, hysteresis is found to be present even when sputtering in the HIPIMS region, therefore, a closed loop control is used to maintain the peak current during the pulse. Variations in peak current as well as the addition of a positive high voltage pulse are examined. Material properties such as crystal structure, morphology and mechanical properties will be presented.

# Target poisoning during CrN deposition by mixed high power impulse magnetron sputtering and unbalanced magnetron sputtering technique

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Target poisoning phenomenon in reactive sputtering is well-known and has been studied in depth over the years. There is a clear agreement that this effect has a strong link on the quality, composition, properties and pronouncedly on the deposition rate of PVD coatings. With the introduction of IPVD techniques such as the relatively novel High Power Impulse Magnetron Sputtering (HIPIMS), which have highly ionized plasmas of the depositing species (metal and gas ions), target poisoning phenomenon is highly contested and thus have been left wide open for discussion. Particularly there have been contradicting reports on the presence of prominent hysteresis curves for reactive sputtering by HIPIMS. More work is needed to understand it which in turn will enable reader to simplify the coating deposition utilizing HIPIMS.

This work focuses on the study of Chromium (Cr) targets when operated reactively in argon + nitrogen atmosphere and in different ionizing conditions, namely (a) pure HIPIMS (b) HIPIMS combined with UBM and (c) pure UBM (Unbalanced Magnetron Sputtering). Nitrogen flow rate was varied (5 sccm to 300 sccm) whereas the average power on target was maintained around 8KW. Target resistivity vs $N_2$ flow rate curves for these conditions have been plotted in order to analyse the poisoning effect. When only one UBM target was operating target poisoning effect was prominent between the flow rates of 80 and 170 sccm. However it appeared reduced and in nearly same flow rate ranges (90 and 186 sccm) when only one HIPIMS target was operating. When 4 UBM targets were operated, target poisoning effect was evident however expectedly moved to higher flow rates (175 sccm and above) whereas appeared diminished when 2UBM and 2 HIPIMS were running simultaneously. Further, to analyze the effect of actual target conditions (poisoning) on deposition rate and on the properties of the films deposited, commercially widely used Chromium nitride (CrN) coatings were deposited in mixed HIPIMS and UBM plasma and at 5 different flow rate of nitrogen. Detail characterization results of these coatings have been presented in the paper which will assist the reader in deposition parameter selection.

**Key words**: HIPIMS, UBM, CrN, Nitrogen flow rate, target poisoning, PVD coatings.

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# Analysing deposited Au-films on 3D structured aluminum alloy substrates as a function of the HiPIMS parameters

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For a wide industrial sector coatings are indispensable. Physical vapor deposition (PVD), especially Magnetron Sputtering (MS) allows the deposition of thin films of high quality. For that reason it is used for many different industrial applications and also for measurement devices of highest precision. An essential disadvantage of this technology is that due to the Line of Sight (LOS) law deposition is limited in coating complex geometries.

HiPIMS (high power impulse magnetron sputtering) is noted for enabling substantial improvements over conventional MS regarding to the deposited coatings and its properties. Short power pulses in the region of about 50 – 500 µs with very high peak power densities can lead to a high density magnetron plasma with high fraction of ionized species with a considerable impact on conformal coating of 3D structures.

Within this work HiPIMS is used to deposit thin Au coatings on various aluminum alloy (AlCuMgPb) substrate structures. Thorough analysis of the coatings dependent on the specific HiPIMS process parameters pulse frequency and pulse duration was carried out. As a function of these parameters, the samples were investigated to what extend the deposited coatings showed differences in deposition rate, structure and homogeneity. Side walls of trenches and drilled holes with aspect ratios of 1:1 – 1:4, as well as Non Line of Sight (NLOS) areas in undercuts, were examined by scanning electron microscope (SEM) in order to investigate a suspected correlation between thickness uniformity and the peak currents during the high-power plasma pulse. For reference, films are also deposited by direct current magnetron sputtering (DCMS) and compared to films grown by HiPIMS.

**Keywords**: HIPIMS, HPPMS, aurum/ gold, high aspect ratio, Line of Sight

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# Optimization of deposition rate in HiPIMS through the control of magnetic field and pulsing configuration

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In HiPIMS process, the magnetic field strength and unbalance and pulsing configuration are key factors in controlling metal ionization rate and fluxes of sputtered species through the substrate. In this work, the effects of pulsing configuration (pulse duration) and external magnetic field on the deposition rate are presented for ten different target materials (C, Al, Ti, Mn, Ni, Cu, Zn, Mo, Ta and W) sputtered using HiPIMS. The external magnetic field was created with a toroidal shaped permanent magnet placed in front of a strong balanced magnetron. The auxiliary magnetic field weakens the magnetic field strength of the central pole and raises the magnetic field strength of the outer pole of the magnetron, shifting the magnetic field configuration towards an unbalanced magnetic trap. The deposition rates in HiPIMS assisted by an external magnetic field have been compared to those obtained in direct current magnetron sputtering (DCMS) and HiPIMS without external magnetic field, under the same experimental conditions (average power, gas pressure). It was found that in the case of HiPIMS assisted by external magnetic field, the deposition rates were approximately 40% to 140% higher compared to HiPIMS without magnetic field and, for some materials, were even higher compared to the DCMS. For Cu and W target materials, time-averaged mass spectroscopy was performed at the substrate position to characterize ion energy distributions and composition of total ion fluxes onto the substrate. Beside higher deposition rates, the HiPIMS assisted by external magnetic field increases the metal ionization rate and significantly improve target utilization (from 18% to 35%) and thin films properties (crystallinity, roughness, hardness and elastic modulus). Experimental results show that deposition rate and coating properties could be optimized by using an appropriate magnetic field and pulsing design.

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# Impact of growth defects on the corrosion behaviour of CrN/NbN coatings deposited by HIPIMS/UBM

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The enhanced corrosion and wear resistance properties of nanoscale CrN/NbN PVD coating have made it a potential candidate for various applications including industrial and biomedical devices. However it has been shown that the growth defects generated during the coating deposition can degrade the coating performance which is undesirable.

In this work CrN/NbN multilayer coatings were deposited using combined high power impulse magnetron sputtering (HIPIMS) and unbalance magnetron sputtering (UBM) techniques.

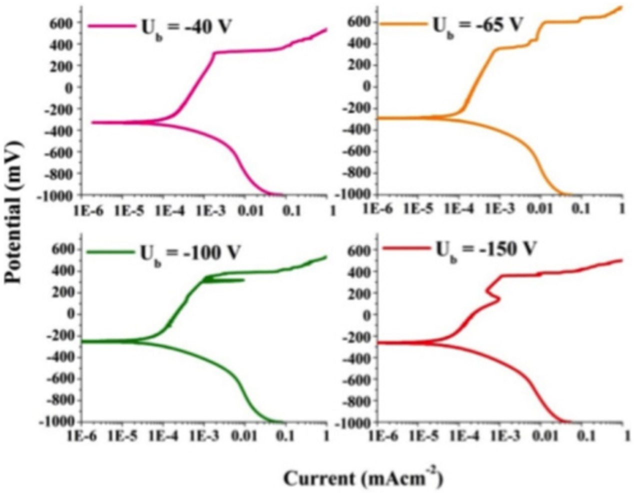
For this study, the substrate bias voltage ($U_b$) was varied from -40 to -150 V whereas the chamber pressure and deposition temperature were kept constant at 0.35 Pa and 200°C respectively.

To study the influence of substrate bias voltage on defect formation and their impact on the coating morphology and properties XRD, SEM, AFM, optical microscopy and potentiodynamic polarisation analytical techniques were used. The crystal structure of the coatings was determined by XRD. SEM was used to analyse the surface morphology of the coatings. SEM images confirmed the formation of four types of defects. Depending on their shapes and growth mechanism, defects were categorised as nodular, open void, cone like and pin hole. The defect density, $A_d$, was calculated by the fraction of the surface area of the sample covered by imperfections. Depending on the variation of the substrate bias voltage, $A_d$ was in the range of 0.0313-0.0430.

The corrosion performance of the coatings was investigated by potentiodynamic polarisation measurements in 3.5% NaCl solution. These measurements revealed that the pitting potential of the coating deposited at -40 V $U_b$, was the lowest (308mV), whereas coating deposited at higher bias voltages had a similar but higher pitting potential (353 mV to 355mV).

This indicates that controlling the bias voltage is an effective tool for coating densification.

These results show that the increasing defect density was counteracted by increased densification of the coatings for higher bias voltages.

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# Highly Ionized Deposition of CrN using MPP

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In different industrial application CrN is used because of its high hardness, corrosion resistance, and high temperature stability. Deposited CrN films using Modulated Pulse Power (MPP) on a lab scale sputtering plant without additional heating and bias voltage already showed higher hardness compared to the state of the art sputtering processes.

Basing on the lab scale static investigations the process was up-scaled to cathode size of approx. 0.5 m length. This presentation will discuss the results gained by both, planar and cylindrical cathodes. Influence of gas composition and peak current on adhesion, hardness, and composition of the films will be presented. In contrast to conventional magnetron sputtering film hardness of 2500 HV can be realized without any additional heating or substrate biasing.

**Keywords**: Hard coatings, Modulated Pulse Power, Rotatable, CrN

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# Investigations on spoke behaviour in reactive HiPIMS with ICCD measurements and energy and time resolved mass spectrometry

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Travelling ionisation zones in HiPIMS plasmas, so-called spokes, have been a topic of interest in the community for several years. Recent research indicates that spokes are responsible for the existence of high energetic ions. In this study we investigate the influence of reactive species on the amount of the spokes and their appearance.

ICCD camera images of spokes were taken for peak discharge currents ranging from 30 A to 80 A were done with reactive gas partial pressures from 0% to 100%, while keeping the total pressure constant at 0.5 Pa. All experiments were done with a 2” chromium target with the working gas argon and the reactive gas nitrogen. The pulse length was 150 µs and the frequency 4 Hz. For each discharge condition 200 images were taken. These images were categorised by a data driven method to find the most probable spoke configuration for given discharge parameters. No filters were used when taking the images, thus the total light intensity was measured by the camera.

For non reactive conditions chromium spokes are well localised with clear boundaries. When reactive gas is added to the discharge blurring of the spokes as well as an increase of the spoke number is observed. Furthermore the hysteresis effect is reduced for higher currents.

The ICCD measurements were used to find interesting working parameters for time resolved ion energy distribution function measurements. Chromium, argon and nitrogen ions, as well as molecular nitrogen ions were investigated by means of time and energy resolved mass spectrometry. Possible correlations between the optical appearance of spokes and the ion energy distribution functions will be discussed.

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# Current-voltage characteristics of a dc magnetron with a hot target

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The high power sputtering technology has great advantages for the synthesis of metallic coatings. One of the variations of this technology is a DC sputtering of a “hot” metallic target in an argon environment. In this case, due to the intense ion bombardment and the controlled cooling of the target, it can be heated up to the temperatures close to its melting point.

During the sputtering of a “hot” metallic target in a glow discharge certain effects that change the current-voltage characteristics (the *I-V* characteristics) of the discharge may occur. They significantly differ from the *I-V* characteristics of a magnetron with an intensely cooled target. The study of the *I-V* characteristics of plane magnetrons with copper and titanium targets of 120 mm in diameter has been carried out in this work. The optical emission spectroscopy was used in order to observe the emission spectra of the discharge during the current-voltage characteristics measurement. As an example, figure 1 shows the experimental *I-V* curves of the magnetron with the “hot” target measured at different pressures. The same curves for the magnetron with the cold target are also presented for the juxtaposition. The plots show that at a certain discharge power density the voltage starts to abruptly increase. These curves show the similar behavior for the titanium target.

The current saturation regime of the discharge during the significant increase of the voltage is most likely caused by the stack effect of the gas close to the target. In this case, this effect appeared due to the heating of the gas not only by the intensive flow of the metal but also, probably, by the heat of the target, since at pressures 1–10 mTorr the gas manages to retain its thermal conduction.

The stack effect of the gas has been confirmed during the analysis of the emission spectra of the discharge. At low power density values, the intensity lines of the neutral Ar atoms and metal ions have increased in the same manner as in the case of a cold target. The transition of the magnetron into the “hot target” regime (the current saturation) has led to a proportional decrease of the intensity lines of argon and an exponential increase of the corresponding lines of the ions of metal.

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

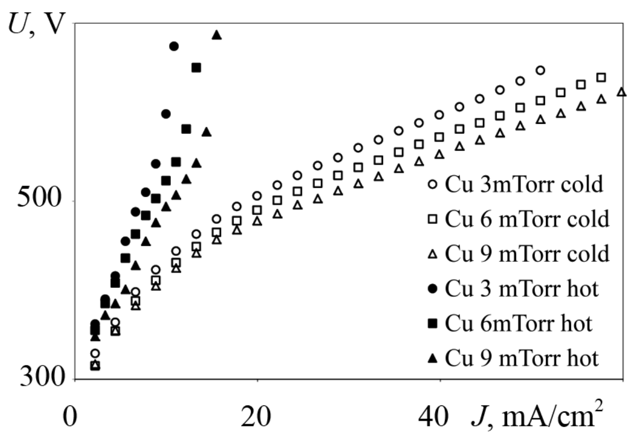


Рис. 1. ВАХ магнетрона с холодной и горячей медной мишенью

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# The model of nitride films deposition using the high power reactive magnetron sputtering technique

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High power reactive magnetron sputtering of metallic targets in an Ar/$N_2$ environment is a very perspective method for the synthesis of nitride films.

The main feature of every reactive sputtering technology is the fact that at certain conditions instabilities occur in the discharge. These instabilities spontaneously transfer the sputtering system from one stable state to another. The hysteresis effect is another important characteristic of the reactive sputtering technique. During the high power sputtering, the heating of the target surface also affects the process, since the target can be heated up to its melting point. The heating of the target is achieved either by sending high power microsecond impulses to the target, or by the fact that the heat outflow from the target occurs at low speeds. In this case, the flow from the target surface is formed not only due to the sputtering itself, but also due to the evaporation. The process becomes more complicated as the ion-electron emission is supplemented by the thermal emission.

For the effective implementation of all of the possibilities of the high power sputtering technology, its common characteristics must be established, and the connection between the dependent and independent variables must be revealed. In this work, a physico-chemical model is offered for the analysis of the nitride films deposition process. The analysis of the processes occurring on all of the surfaces of the sputtering system (target, substrate, walls) has been carried out. The target plays an important role, since the nitride film appears on its surface as a result of a chemical reaction, the sputtering and the heating of the target take place due to the ion component of the discharge current. The evaporation also takes place. The target surface is the source of electrons sustaining the glow discharge. The stationary state of the target surface is described by the equation based on the main laws of chemical kinetics, the Arrhenius equation and the Langmuir isotherm.

Two other surfaces are passive. The nitride film forms on top of them due to the flow of atoms and molecules coming from the target. Their stationary state is also described by the algebraic equations. The full analytic description of the reactive sputtering process is made using the system of eight equations including the gas balance equation and the equations describing the inflow of $N_2$ to all of the surfaces and the work of the vacuum pump.

The main independent parameters in this system of equations are nitrogen content and the discharge current. The numerical solution of the system allows us to determine all of the dependent variables of the sputtering process, which include the nitrogen partial pressure and its content on every surface. Besides the analysis of the effect of the independent variables on the chemical composition of every surface is possible. The performed calculations showed that all of the indicated relations represent the S-like curve, which allows the hysteresis effect. The juxtaposition of the results of this research and the results obtained from the previously developed model of the DC reactive sputtering of a cooled target is presented in this work.

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

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# Corrosion behaviour of post-deposition polished droplets-embedded arc evaporated and droplets-free HIPIMS/DCMS coatings

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## Abstract

In this study, the effect of metal rich core of the droplets on the corrosion properties of TiN, CrN and ZrN arc evaporated nitride coatings has been investigated and the corrosion properties of such coatings have been compared with droplet free, highly dense coatings grown by combined high power impulse magnetron sputtering (HIPIMS) and direct current magnetron sputtering (DCMS) technique. An industrial size Hauzer HTC 1000-4 system enabled with HIPIMS technology was used for the deposition of combined HIPIMS/DCMS coatings. The corrosion behaviour of the coatings was studied by potentiodynamic polarisation test (ACM instruments Gill AC potentiostat, -1V to +1V) using 3.5% NaCl solution. Initially, as-deposited arc evaporated coatings with an exposed surface area of 1 cm2 were subjected to corrosion. Then, the coatings were gently polished to expose the metal rich core of the droplets. Subsequently, fresh un-corroded area of the polished coating was subjected to corrosion with previously corroded area masked. It has been found that mechanical polishing considerably deteriorated the corrosion performance of arc coatings by forming more than one galvanic couple between the two parts (metal rich, nitrogen rich) of the same droplet itself or between the metal rich part of the droplet and the adjoining coating. It has been further demonstrated that the droplet free highly dense HIPIMS/DCMS coatings exhibited superior corrosion resistance as compared to the arc-evaporated coatings. Raman analysis was used to study the constituents of the corrosion products. Scanning electron microscopy (SEM, planar view) was used to examine the as-deposited and corroded coating surfaces to define morphological differences. Energy dispersive X-ray (EDX) analysis was done to study the composition of the coatings.

# The influence of the reactive gas flow on the structure and properties of TiAlCN/VCN films

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Reactive sputtering in PVD processes can be utilised to fine tune the properties of the coating microstructure. The microstructure of the coating affects mechanical properties such as hardness and friction coefficient.

The current research investigates the transformation of the microstructure and mechanical properties of TiAlCN/VCN coatings deposited by combined HIPIMS and UBM technique, with varying reactive gas flows of nitrogen and methane.

Semi-quantitative EDX analysis of the coatings showed a linear increase in nitrogen content with total reactive gas flow. Unlike nitrogen, carbon did not exhibit correlation between reactive flow and content in the coatings. Raman analyses indicated that the intensity ratio of carbon D/G peaks increased continuously with reactive gas flow.

Increasing the reactive gas flow from 40 to 85 sccm increased the hardness of the coating from 780 up to 3721 HK. Further increase to 95 sccm resulted in hardness reduction associated with the appearance of columnar structure as well as reduced deposition rate.

The structural characteristics and crystallographic phase of the coatings were determined by scanning electron microscopy and X-ray diffraction using both Bragg–Brentano and glancing angle parallel beam geometries. For low reactive gas flows the microstructure is dense, with a glossy amorphous morphology and consisted of a metal-rich phase. When the flow is increased, TiAlCN/VCN coatings grow in a NaCl-type cubic crystalline phase with a microstructure which is dense and with grains that are randomly oriented. A reactive gas flow of 95 sccm resulted in a morphology consisting of large diameter (~160 -300 nm), well defined columns, terminated with sharp triangular tops, resulting in a faceted topology and a cubic phase. Further increase of reactive gas flow up to 105 sccm led to a cauliflower-like surface morphology with column sizes of 50-200 nanometres and a (220) preferred orientation.

We have demonstrated that wear resistant, low friction TiAlCN/VCN coatings form a different microstructure depending on the reactive gas flow. This can be used to obtain better tribological properties of the TiAlCN/VCN coating as well as finding new applications

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# Effects of charge voltage on fabrication of AlCrN coatings by a high power impulse magnetron sputtering

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## Abstract

In this study, AlCrN coatings were deposited on WC-Co substrates by high power impulse magnetron sputtering (HIPIMS). The charge voltages of the HIPIMS power were varied from 350V to 550V so that different peak voltages were applied on the AlCr target during coating deposition. The coatings were characterized by utilizing X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), nanoindentation, and pin-on-disk tribometer. The results indicated that all the AlCrN coatings exhibited (Al, Cr)N solid solution phase with FCC NaCl-type structure. With increase of discharge voltage, the preferred orientation shifted from (111) to (200). A diffraction peak of h-AlN phase at 2θ=~33° was detected at charge voltage higher than 500 V. The hardness of the AlCrN coatings increased from 24.9 GPa at charge voltage of 350 V to 30.8 GPa at 450 V, and then decreased to 20.6 GPa with further increase of charge voltage to 550 V. During vacuum annealing, no remarkable differences on the coating structure and mechanical properties below 800 °C. When the annealing temperature increased to 1000 °C, changes were observed on the coating phase structure and cross-sectional morphologies. Correspondingly, the coating hardness decreased apparently. The friction coefficient and wear rates of the coatings against $Al_2O_3$ balls also exhibited some relationship with the charge voltages and test temperatures. The inherent mechanism was analyzed and discussed.

**Keywords**: High power impulse magnetron sputtering (HIPIMS); Charge voltage; AlCrN coating; Microstructure; Tribological behavior

# Direct metallization of plastics by HIPIMS

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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.

Investigations on different plastics (PPSU, PEI, PEEK, PESU, PSU, PMMA) showed significant improvement of coating adhesion by only using direct metallization without any prior treatment. Tape test and combined cross cut and tape test was applied for characterization of the adhesion. The improved adhesion is influenced by the process parameters, mainly the ionization during the process, characterized by the peak current density.

# Study of the effects of HIPIMS pulse packages on the peculiarities of carbon nanotubes creation during the growth of diamond like carbon composite matrix

Dr. Vachagan Meliksetyan

The main objective is the development of innovative composite material possessed by both high electro and thermo conductivity. The surrounding DLC matrix guarantees good mechanical stability. However, the problem of combination of the both parts seems very complicated and requires great efforts which may be solved only by a comprehensive approach. Creation of unique composite material comprising carbon nanotubes (CNT) incorporated in a diamond-like carbon (DLC) matrix is really breakthrough for material science. We offer the innovative composite material consisting preferable of carbon atoms with a mixture of microstructures whose synergy will allow addressing various goals. The carbon nanotubes (CNTs) being embedded in a diamond like carbon matrix will allow obtaining high electrical and thermal conductivities and corresponding functionalization of nanotubes what may assist in using the various specific features. It is proposed to grow the composite films by plasma-enhanced chemical vapor with plasma containing various additives such as for example Fe, Co, or Ni, facilitated the formation of CNT and thus coatings with optimal features. The mentioned additives may be inserted by using Hipims technology The presence of nanotubes leads to sharp increase of charge carrier's mobility and, respectively, conductivity by up to many orders of magnitude. The innovated technology allows obtaining the DLC matrix with incorporated CNTs served as additional conducting channels without deterioration of mechanical and optical properties of coatings. If anybody will succeeded in finding the way of insertion nanotubes with optimal parameters into bulk of surrounding matrix it may be real breakthrough not only for decision the considered task but also for many other problems of material science. It is well-known that insertion of even nonessential number of CNT capable to increase the conductivity of composite material on many orders of magnitude To achieving percolation threshold it is important to enhance CNT concentration; however the percolation probability depended also on a number of other factors and in first place on geometrical dimensions of CNT and on probability of transferring between various nanotubes.

# Electrical conductive yarn and fabric obtained by using R2R-HIPIMS

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## Abstract

General trend in the development of high quality electrically conductive fabrics and yarns for smart textile application such as healthcare and sport products have attracted a significant attention in recent year. To meet this demand, conductive brass-deposited thin film on both polypropylene (PP) non-woven fabric and yarn have been investigated by using the Roll-to-Roll high power impulse magnetron sputtering (R2R-HIPIMS) system, in which HIPIMS technique provides high plasma density for deposition at low substrate temperature, hence the obtained coating is expected to present better than those conventional processes.

Experimental results show that a uniform brass coating can be deposited with highly crystalline face-centered cubic alpha structure. The substrate can be without damage below a specific cathode peak power of usage that corresponds to a given linear feed speed of the fabric and yarn. Ultimately, in the case of PP non-woven fabric, it can be deposited safely with a target peak power density of 276 $W/cm_2$ at a maximum linear feed speed of 1.5 m/min. This corresponds to an average film thickness of 65 nm covering over the PP non-woven fabric and gives a sheet resistance of 501 (Ω/□). Further lower sheet resistance can be achieved through repeated coating runs, for example from 501 (Ω/□) to 88 (Ω/□) respectively from one coating run to four times coating runs. Based on these features, the brass-deposited textiles may have a great interest for the application on the smart textile industry.

**Keywords:** Electrically conductive textile, $Cu_{65}Zn_{35}$ brass, High power impulse magnetron sputtering, Roll-to-Roll coating.

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# High-rate reactive high-power impulse magnetron sputtering of Hf-O-N films with tunable composition and properties

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High-power impulse magnetron sputtering with a pulsed reactive gas flow control [1, 2] was used for reactive depositions of Hf-O-N films with tunable composition and properties. 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 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. A pulsed reactive gas ($O_2$ and $N_2$) flow control made it possible to produce high-quality Hf-O-N films of various elemental compositions with high deposition rates of 175 - 240 nm/min. All films were nanocrystalline and their elemental compositions were varied gradually from $HfO_2$ to HfN. We present the gradual change of hard (18 GPa), highly optically transparent (extinction coefficient of 5x10-4 at 550 nm) and hydrophobic (water droplet contact angle of 101°) $HfO_2$ films into harder (25 GPa), opaque and more hydrophobic (water droplet contact angle of 107°) HfN films.

**Keywords**: Reactive HiPIMS; Pulsed reactive gas flow control; Hf-O-N films; Tunable properties

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