

Fifth International Conference on HIPIMS *Abstract Book*









The potential structure of ionization zones in high power impulse sputtering

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Over the last years it has been established that traveling ionization zones are common in high power impulse magnetron sputtering. They are plasma instabilities which seem to be related to similar instabilities observed in other ExB discharges, such as Hall thrusters. Fast photography is a preferred experimental approach as it is not disturbing the plasma as probes do, for example. Fast cameras of two types are employed: a short-exposure frame camera, and streak camera. Images show the concentration of ionization in narrow zones, and the formation of plasma flares (e.g. [1, 2]). Species-selective ion energy measurements are complementary as they provide entirely different information. This contribution summarizes and unifies different aspects from different experiments related to ionization zones. The most consistent interpretation of the observation can be achieved by assuming that each ionization zone is associated with a potential hump of typically several 10 V height [3]. The formation of such humps follows naturally from the different inertia of electrons and ions, as electrons can leave the ionization zones faster, leaving a slight positive excess charge behind. The formation of a potential hump implies that the hump is enclosed with an electrical double layer. Double layer formation between plasmas of different properties (e.g. density) is well known in plasma physics, and should be expected. Double layer formation was proposed by Brenning et al. [4], however, they considered only one side of the zone. A more complete description requires that the integral over the potential gradient along the racetrack is zero. In other words, the potential needs to be the same when electrons execute a closed drift to the same location over the racetrack [3]. The double layers may not be symmetric - only the reproduction of the potential is required. This is illustrated in Fig. 1. A number of consequences arise, most importantly an energy enhancement for ions returning the target, and the possibility of ions to escape the near-target region where, on average, the electric field points towards the target. Yet another consequence is the asymmetry of charged particles leaving the near-target region: ions on the side traveling with the ionization zone can "surf" the potential hill, thereby gaining higher energy than ions going in the other direction [5]. The effect can be substantial as energetic features in ion energy distributions have been found that reach up to several 100 eV. We conclude that ionization zones and their positive potential humps and double layers are essential, critical features for the operation of the discharge and properties of particle fluxes.



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Fig. 1 Positive potential hump V, consistent electric field E, and net charge distribution (from Ref. [3])

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On the HiPIMS benefits of multi-pulse operating mode

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High Power Impulse Magnetron Sputtering (HiPIMS) has reached nowadays its maturity penetrating the industrial processing. The main interest of this technology concerns the improved control of thin films properties [1] due to the important fraction of ionized sputtered material flowing towards the substrate.

Besides its high ionization efficiency, HiPIMS has two major drawbacks: (i) the lower deposition rate, compared to Direct Current Magnetron Sputtering (DCMS) and (ii) the difficulty of operating at very low pressure, typically below 0.4 Pa. To overcome these points, different approaches have been recently proposed [2, 3].

Our focus is the optimization of the HiPIMS process with respect to DCMS, based on the physical understanding of the plasma state when using sequences of pulses, instead of general single high power pulse. Operating at standard frequencies (~ 1 kHz), the duty cycle is kept low (0.5 to 5.0 %), consecutive single pulses as well as consecutive sequences of short multi-pulses can be considered as independent of each-other. The contribution compares the plasma behaviour between single-pulse and multi-pulse sequences, separated by long afterglows and generated with the same power unit [4]. HiPIMS process is improved for multi-pulse mode as proved by the temporal evolution of the ion current to the substrate, correlated to optical emission and absorption signals of the sputtered species and deposition rate.

Controlling the temporal sequence of pulses, parameter by parameter, the multiple benefits of this approach are highlighted. The temporal sequence can be very general, but it also can be reduced to the chopped HiPIMS [2] (for periodic sequences), or managed as single short-length pulses, limiting thus the metal back-attraction. This approach permits also non-periodic sub-sequences within a sequence of pulses.

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Impact of coating defects on the corrosion protection capabilities of TiN films deposited by dc-magnetron sputtering and HIPIMS by a novel scanning method

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Abstract:

Coatings deposited by magnetron sputtering have been characterised for decades for their corrosion protection capabilities. The film density has often been stated to be the key factor and improvements were published, mostly based on different electrochemical corrosion measurements. Often discrepancies have been found between the results of these measurements and those of salt spray tests, which mostly were attributed to the impact of coating defects. With the High Power Impulse Magnetron Sputtering (HIPIMS) technology further improvements of the density of sputtered hard and thin coatings could be achieved as published by many research groups. The aim of this work was to examine if these improvements do lead to improved corrosion protection properties of HIPIMS deposited films.

With the Large Area High Resolution (LAHR) mapping a method has recently been developed at the fem which allows localizing and even characterising the coating defects present on a coated sample. It is based on scanning the topography of the entire surface of lab sized samples (several cm2) with a lateral resolution in sub-um range by confocal microscopy. The defect data are generated by complex custom-built templates based on Mountains Map™ and Visual Basic[™]. Reliable defect statistics, defect maps as well as coordinates for each specific defect are available for the first time with this method.

2.5 µm thick TiN-films were deposited on polished steel samples using dc-magnetron sputtering and HIPIMS and afterwards exposed to neutral salt spray test. Using LAHR-mapping the defect structure of the entire sample surfaces was characterised and by scanning the same samples before and after the corrosion test each corrosion site could be traced back to its source. With this the coating defects responsible for corrosion could be identified. The results of these tests as well as the defect statistics data for several defect types and sizes are presented and discussed with respect to their impact on the corrosion behaviour of the coated samples. Additionally the origin of these defects will be focused.

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15 Year of HIPIMS – a success story

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Even though the history of high power pulse magnetron sputtering HPPMS or high power impulse magnetron sputtering HIPIMS can be tracked back to the 1960', the community refers to the paper from V. Kouznetsov et al. in 1999 as the seminal publication for this technology. Within the following 15 years HIPIMS has paved its way from academic laboratory to industrial applications.

This talk will show the development from the early works and the topics proposed towards industrial processes today. Examples will be given on trench filling, interface modification, and advanced coatings with respect to properties and deposition rates. Starting with fundamental investigations and proposed properties the development towards industrial processes today will be reviewed. Parallel development in the field of pulse generation and the available state of the art power supplies will be addressed.

High-rate reactive high-power impulse magnetron sputtering of hafnium dioxide films

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High-power impulse magnetron sputtering with a pulsed reactive gas (oxygen) flow control [1] was used for high-rate reactive depositions of densified stoichiometric hafnium dioxide films. The depositions were performed using a strongly unbalanced magnetron with a planar hafnium target of 100 mm diameter in argon-oxygen gas mixtures at the argon pressure of 2 Pa. The repetition frequency was 500 Hz at the average target power density from 5 Wcm-2 to 54 Wcm-2 during a deposition with duty cycles from 2.5% to 10%. The substrate temperatures were less than 165°C during the depositions of films on a floating substrate at the distance of 100 mm from the target. Usual deposition rates, being around 15 nm/min, were achieved for the target power density of 5 Wcm-2. An optimized location of the oxygen gas inlets above the target and their orientation to the substrate surface made it possible to improve quality of the films due to minimized arcing at the sputtered target and to enhance their deposition rates up to 345 nm/min for the hafnium dioxide films at the target power density of 54 Wcm-2 and the duty cycle of 10%. This deposition rate was about 75% of that achieved for pure hafnium films prepared under similar conditions. The hafnium dioxide films were crystalline with a monoclinic phase. They exhibited hardness of 15 - 18 GPa, refractive index of 2.06 - 2.12 and extinction coefficient of 0.0001 - 0.001 (both at the wavelength of 550 nm).

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Poster session preferentially

Time-resolved study of simultaneous combination of **HiPIMS and mid-frequency pulsed dc discharge**

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Integration of HiPIMS Equipment into an Industrial Coating **Production for Cutting Tool**₅

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Abstract

HiPIMS coatings are rapidly gaining ground for cutting tool applications due to advantages such as smooth, droplet free coatings with superb adhesion and perfect homogeneity all around the tool geometry. Hence, more and more cutting tool producers are in the process of integrating HiPIMS coating machines into their production.

This paper will discuss the full process chain of an industrial coating production - starting with jigging, surface and cutting edge preparing, cleaning, coating and finishing operation - dedicated to the HiPIMS coating process. Advances of the HiPIMS deposition equipment will be presented as well as specific modifications of the auxiliary processes prior and after coating.

Thick PVD coatings are known to increase the tool life for milling operations with indexable inserts in cast iron. Crank shaft milling in the automotive industry is a very typical mass production example. The paper will introduce the design and the performance of 6 µ and even 9 µ thick HiPIMS coatings done in industrial mass production.

A novel AITiSiN film deposited with HiPIMS will be presented as a model system for all the steps of a commercial coating production. The evaluation will include film characterisation and recent cutting test results.

Keywords: HiPIMS, Sputtering, Cutting Tools.

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Description of HiPIMS plasma regimes in terms of composition, spoke formation and deposition rate

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The behaviour of Cu and Cr HiPIMS (high power impulse magnetron sputtering) discharges was investigated for a broad range of the current-voltage characteristic curves. Characterization at the near-target region was done by optical emission spectroscopy and optical imaging; and at the substrate position by quartz microbalance and energyresolved mass spectrometry. We found that the inflection points typical of HiPIMS current-voltage characteristics allow identifying plasma regimes perfectly differentiated in terms of deposition rate, flux composition of species at the substrate, and the nature of plasma self-organization [1]. In particular, the reorganization of plasma into spokes (welldefined areas of high ionization) was associated to a regime of high plasma conductivity, where also deposition rate is limited. The spoke-dominated regime disappears at higher powers, where plasma emission becomes homogeneous. This homogeneous regime is associated to an increase of deposition rate, which seems to be due basically to an increase of metal neutrals and metal double-charged ions. The relevance of secondary electron emission mechanisms for the support of the spoke-dominated regime in reactive and non-reactive sputtering conditions will also be discussed.



Up, left: Voltage-current curve built from the output voltage value of the power supply and the target current value at a time of $t=180 \mu$, for a Cu discharge. Down, left: Current pulses of the same discharge. Right side: Frontal imaging of the discharge corresponding to the points A, B, C, marked in the voltage-current curve.

Determination of sputtered species densities in HIPIMS discharge by optical emission spectroscopy

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Developing of magnetron sputtering process is aiming mainly to increase ionization of sputtered particles, to improve target yield or to enhance deposition rate. HIPIMS discharge yields higher number density of ionized sputtered species than DC case. However, it is difficult to estimate absolute number densities of the sputtered species (atoms and ions) in ground and metastable states from optical emission spectroscopy, because the ground and metastable levels do not directly produce any optical signal. There has been developed variety of indirect methods, however, mainly for rare gases to obtain information about number densities of metastable levels. Our research aim is to adapt these techniques such as self-absorption or effective branching fraction [1] on conditions of HIPIMS discharge. We ran experiments on magnetron with 20 cm titanium target. Lines of titanium neutrals and ions which originate at same higher level and fall to certain sub-level of neutral or ion ground state were carefully selected in our research. The population of these ground state sub-levels was assumed to follow Boltzmann distribution. Fitting theoretically calculated branching rations to experimentally measured ratios of the line intensities enable us to determine number densities of ground state sputtered species from optical emission spectroscopy in a HIPIMS discharge - see estimated evolution of Ti ion number-density together with the discharge current at Fig. 1. This research has been supported by the CZ.1.05/2.1.00/03.0086 and GACR P205/12/0407 projects.



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Fig. 1:

Time resolved number density of Ti ions and discharge current for 1200 µs HIPIMS pulse.

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Regime transition in HiPIMS: volume averaged and phenomenological models.

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HiPIMS discharges evolve through a range of regimes, depending on the plasma composition and spatial organization. The transition zones between the regimes correspond to dierent slopes in the Voltage-Current characteristics [T de Los Arcos et al., J. Phys. D: Appl. Phys., 335201 (2013)], and therefore to dierent equivalent conductivity of the plasma. By means of a uid and a kinetic model, we reproduce both a low (lpeak $\approx 5.10^{-2}$ A/cm²) and a high current (lpeak ≈ 5 A/cm²) discharge. We discuss the applicability of a fluid approximation by comparing the electron energy distribution function (EEDF) obtained with the kinetic model with a Maxwellian EEDF at the temperature yielded by the fluid model. Moreover, we analyze the plasma composition throughout the pulse, commenting on the in influence of doubly charged ions [A Hecimovic et al., J.Phys. D: Appl. Phys., 095203 (2008), C Maszl et al., arXiv:1311.7545v2]. In the high current case, the plasma spatial organization in rotating structures is modeled by means of an additional gas source. Finally, we look at the overall discharge and we set up a global lumped circuit model composed of non linear resistors. This phenomenological model allows us to describe the time varying V-I curve, spanning the dierent regimes.

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Figure 1

On the left, the magnetron set up. On the right, the evolution of the voltage (black solid line) and the discharge current (blue dashed line) during the pulse. The green and red solid lines give the time varying output current obtained with the kinetic model, without and with the eect of enhancing Ar gas diusion.

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Reactive Process Control of HIPIMS Discharges

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Reactive sputtering is a well-established technology for industrial sputtering. The process stabilization is carried out by varying the average power or the reactive gas flow. As input parameters the partial pressure of the reactive gas, the target voltage or the plasma emission is commonly used.

For reactive HIPIMS challenges regarding process control arise. For instance the change of discharge voltage is strongly depending on the capacity of the used HIPIMS power supply. Even the plasma emission is lower in HIPIMS discharges (time-averaged) since the duty cycle is normally in the lower percentage range.

This talk will give an overview of different approaches for a reactive process control in combination with HIPIMS. The discussed feedback systems are based on plasma emission monitors with either optical filters for single emission lines or with a spectrometer. The controllers are regulating the oxygen flow by piezo-valves or mass flow controllers or by changing the off-time and therefore the average power.

The different approaches will be presented for Alumina on different sputtering plants equipped with planar magnetrons or even rotatables. The results include the voltage and current characteristics as well as the deposition rates and selected film properties.

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Abstract Book

The influence of substrate-bias on the HIPIMS Tungsten films

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Nowadays, tungsten is considering one of the most attractive materials to deal with their protective role for first wall applications in the nuclear fusion reactors [1-3] due to its properties: low sputtering yield, low-activation, high melting point, high thermal conductivity, and low thermal expansion. On the other hand, because of their specific properties (enhanced radiation-resistance and adhesion strength), nanostructured materials are being very interesting for fusion applications. One of the main concerns in a reactor environment is the structural stability and the adhesion to the substrate.

In this work, we report on the growth of nanostructured tungsten films with a thickness in the micro-meter range by using high impulse power magnetron sputtering (HIPIMS) on different substrates (silicon and steel). We aim to study the W coatings morphology, microstructure and stress state in two deposition modes: a) grounded substrate and b) bias voltage applied to the substrate.

Samples were characterized using scanning electron microscopy (SEM) and X-ray diffraction (XRD). The influence of the substrate-bias on microstructure, film texture and film stress-state is discussed.

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Investigation of plasma parameters during the HIPIMS deposition of semiconductor oxide thin films for water splitting application.

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A high power pulsed magnetron sputtering system HIPIMS was investigated as a source for the reactive sputtering of various oxide semiconductor thin films. Fe2O3 and WO3 thin films were deposited on glass substrate with FTO electrode and on pure quartz glass by this method. Pure iron and tungsten targets were reactively sputtered in Ar+O2 working gas mixture. Depositions were done without external heating of the substrate during the deposition process. Deposited films were characterized as deposited and after annealing process. Concentration of oxygen in the plasma was varied during the deposition process and influence on parameters of deposited films was investigated. A pulsed modulated RF bias synchronized with HIPIMS pulses was applied on the substrate during the deposition. The frequency of the RF generator used for the bias was 13.56 MHz. This pulsed modulated RF voltage induced pulsed DC bias around the substrate with controllable amplitude in the range 30V - 100 V. Synchronization of the modulated RF voltage was controlled in the way that induced pulsed DC bias was present at the substrate only at defined time during the HIPIMS pulse when ionized sputtered particles were present in the plasma. Plasma parameters were investigated at these conditions by means of time resolved Langmuir probe. Ionization degree of depositing particles was measured with modified biased QCM monitor with magnetic electron filter. Ion flux on the substrate was monitored by high frequency ion flux monitor. The influence of these plasma parameters on deposited films parameters was investigated.

Langmuir probe measurements of negative ion densities in reactive HiPIMS discharges

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Using a Langmuir probe, the temporal evolution of negative oxygen ion densities in the afterglow of a HiPIMS discharge has been determined. The discharge was operated with a titanium target in a range of argon-oxygen gas mixtures $(O_2 \text{ partial pressures } p_{O_2} / p_{\text{total}} \text{ of } 0.1, 0.3 \text{ and } 0.5)$, and with the following parameters: power $P_{\text{avg}} = 100 \text{ W}$, frequency f = 100 Hz, and pulse width $_{on}$ = 100 μ s.

The electronegativity, (=n-/ne), was observed to monotonically increase during the discharge afterglow for all three oxygen partial pressures up to peak values of = 19, 316 and 396 for p_{O2} / p_{total} = 0.1, 0.3 and 0.5, respectively. Negative ion density, n₋, was observed to decrease in the afterglow with long characteristic decay times: $\rho_{r} = 1600 \, \mu s$, 525 μ s and 315 μ s for p_{O2} / p_{total} = 0.1, 0.3 and 0.5, respectively. The O- density n₋ was found to be relatively constant during the initial afterglow with values of approximately $n \approx 1 \times 10^{15} \text{ m}^{-3}$.

It is proposed that the increased electronegativity for increasing oxygen partial pressure in the discharge is due to a larger number of high-energy metastable oxygen molecules, O_2^M , being formed during the on-phase due to higher oxygen availability. Results are discussed in the context of diffusive wall losses as well as volume formation and destruction mechanisms of both negative ions (O-) and metastable oxygen (O_2^{M}).

Comparison of plasma diagnostics and materials characterization of Ni and NiO thin films deposited by reactive DC and HiPIMS discharges.

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Abstract

Metal transition oxide has attracted a great deal of attention since the last decade, among them, Nickel Oxide is considered to be a reference for transparent p type semiconductor. Numerous methods to deposit NiO thin films have been used, among them, Direct Current (DC) reactive magnetron sputtering is considered to be the most widely used technique. Recently, our team synthesized these films with HiPIMS technique, where the electrical power is delivered during a short time, less than hundreds of µs. They showed that optical properties can be managed with pulse width duration more accurately. Despite the large number of research, to our knowledge, a comparison of NiO films properties deposited under both techniques has never been reported.

Therefore, in this communication, we will establish the link between the plasma and the thin films properties deposited by reactive DC and HiPIMS. The first part will introduce the result of plasma characterization obtained by optical emission spectroscopy (OES) measurements for both discharges. OES measurements have been performed in time averaged and time resolved mode at 3 cm above Nickel Target for HiPIMS. In a second part, results from thin films characterization will be showed. The deposition of NiO films (100 nm and 300 nm) have been performed on glass, Si and ITO substrates. The influence of oxygen partial pressure on the structural, microstructural, compositional, optical and electrical properties of NiO films was investigated by X ray diffraction (XRD, scanning electron microscopy (SEM), Atomic force microscopy (AFM), X ray photoelectron spectroscopy (XPS), spectroscopic ellipsometry and four probes method.

On periodic oscillations observed on cathode voltage and discharge current during HIPIMS process

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HiPIMS is promising technique for depositing thin layers. Instabilities accompanying HiPIMS also known as spokes were firstly observed independently by Anders at al [1] and Kozyrev et al [2]. Since then, OES and Langmuir probes are usually used for spoke diagnostics [3]. In our research, periodic oscillations were not only observed on OES, but also on cathode voltage and discharge current. Alcatel SCM 650 sputtering system equipped with 20 cm titanium target and balanced magnetic field configuration was employed. Melec SIPP 2000 HiPIMS generator with maximum allowed voltage of 1000 V and maximum allowed current of 500 A was used. The generator was operated in constant voltage mode; discharge duration was 200 µs with repetition frequency of 50 Hz. When the discharge current overreached typically 250 A, the periodic oscillations of discharge current and cathode voltage with frequency around 350 kHz were observed. Amplitude of the current oscillations was around 25 A, amplitude of the voltage oscillations was around 400 V. Varying discharge voltage different current time evolutions were obtained. However, the actual frequency of oscillations depended always on actual discharge current as seen in Fig.1. This behavior is in contrary with behavior observed by Winter et al [3]. Fig. 2 shows that with the increasing pressure the frequency of oscillations also increases. This result agrees with observations of Ehiasarian et al [4]. Aforementioned results lead us to conclusion that observed oscillations of electrical quantities are another, yet not described manifests of the spoke phenomenon.



Fig.1

Actual frequency of oscillations as function of actual discharge current for constant argon pressure of 1 Pa and different applied voltages.



Fig. 2

Actual frequency of oscillations as function of Ar pressure. Data for different voltages are joined together. This research has been supported by the

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Coating solutions and applications, produced by Ingenia -S3p[™], the HIPIMS technology of Oerlikon Balzers

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The unique S3p[™] high power sputtering system opens up several opportunities for coating development and control of coating properties. Using power density pulses from 500W/cm2 to 2000W/cm2 with very high accuracy and pulse duration from 0.05ms to 100ms we can control coating density and reactive gas dynamics in an unprecedented way. For example, with S3p[™] it is possible to suppress the interaction of target poisoning and reactive gas pressure. This effect allows a much better control of reactive sputter processes like TiN, TiO2, ZrN and aluminum oxide. For cutting tools applications we found that in general, the coatings produced by S3p[™] are best in class and beyond for selected applications. Oerlikon Balzers was able to launch a new product series, BALIQ, including the BALIQ TAP ALCRONOS and BALIQ MICRO ALCRONOS coating products.

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On the origin of energetic metal ions during high power impulse magnetron sputtering of titanium

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In direct current magnetron sputtering (dcMS) or any other sputter technique mechanical properties of thin films are influenced by the released energy per incident atom or ion. The connection between particle energies, substrate temperatures and thin film properties are documented in structure zone diagrams [1]. Whereas the possibilities to control the energy distribution function of atoms and hence the mechanical film properties are limited, ion energy distribution functions (IEDF) can easily be tailored via arbitrary biasing. A high ionization degree is therefore beneficial to create a tailored group of high energetic (HE) species.

A high degree of ionization and a high energy group of metal vapor ions occurs naturally in high power impulse magnetron sputtering (HiPIMS) plasmas. Especially, the existence of HE ions is a major difference to dcMS. A power scan from dcMS-like to HiPIMS plasmas was performed, with a 2-inch magnetron and a titanium target as sputter source and argon as working gas. Combined time- and energy resolved ion mass spectroscopy (dt =2 ms) and phase resolved optical emission spectroscopy (dt =1 ms) measurements where used to unravel the origin of Ti+ and Ti2+ ions. It was found, due to the excellent time resolution, that low energy and high energy groups of Ti ions for each charge state coexist at the same time. This implies that both groups have to be created at different locations or by different creation mechanisms within the plasma. Furthermore, the HE group appears only if the plasma is in the spoke regime [2].

A double layer (DL) model is capable to explain the obtained results. If an atom becomes ionized inside the spokes region it is accelerated because of the DL to higher energies whereas its energy remains unchanged if it is ionized outside. Apparently, only if spokes and a DL are present the confined particles can gain enough energy to leave the magnetic trap. Our results corroborate the findings of [3] and motivate why spokes are the essence of HiPIMS plasmas.

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Coating solutions and applications, produced by Ingenia -S3p[™], the HIPIMS technology of Oerlikon Balzers

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The unique S3p[™] high power sputtering system opens up several opportunities for coating development and control of coating properties. Using power density pulses from 500W/cm2 to 2000W/cm2 with very high accuracy and pulse duration from 0.05ms to 100ms we can control coating density and reactive gas dynamics in an unprecedented way. For example, with S3p[™] it is possible to suppress the interaction of target poisoning and reactive gas pressure. This effect allows a much better control of reactive sputter processes like TiN, TiO2, ZrN and aluminum oxide. For cutting tools applications we found that in general, the coatings produced by S3p[™] are best in class and beyond for selected applications. Oerlikon Balzers was able to launch a new product series, BALIQ, including the BALIQ TAP ALCRONOS and BALIQ MICRO ALCRONOS coating products.

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Cr thin films deposited by DOMS (Deep Oscillations Magnetron Sputtering) with a Cyprium III power source.

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Magnetron sputtering technologies are widely used for the deposition of thin films in many commercial applications. In recent years, high power impulse magnetron sputtering (HiPIMS) and modulated pulse power magnetron sputtering (MPP), a variation of HIPIMS, have shown great advantages as compared to the conventional (DCMS) and pulsed dc magnetron sputtering (PMS) techniques. Unlike the simple one pulse shape in HIPMS, MPP generates a high density metal ion plasma by first producing a weakly ionized plasma followed by a transition to a strongly ionized plasma within one overall pulse. However, commercially available HiPIMS plasma generators have not been able to create stable and arc-free discharges in many reactive processes.



Figue 1 I-V characterisitcs for the DCMS and DOMS discharges used in this work.

In this work a new method of generating an arc free discharge for reactive HiPIMS has been used. A Cyprium III plasma generator from ZPulser has been used to deposit Cr thin films by deep oscillation magnetron sputtering (DOMS). Each DOMS pulse consists of a packet of single oscillations. The voltage and current gradually increase to their maximum value (Vp and Ip) during the voltage on-time (ton), and then gradually decay, reaching zero before the end of the oscillations period (T). As shown in figure 1, the resulting discharge is highly ionized with a n exponent calculated from the empirical power law $I = k \times Vn$ well below the typical DCMS values.

The effect of deposition pressure and peak current on the structural (X-Ray diffraction), chemical (Electron Probe Micro-Analysis) and morphological (Scanning Electron Microscopy) properties of the films was studied. All depositions were done at constant average power (1.2 KW) and a constant thickness of 1 micrometer was deposited for all the films. Increasing the peak current at constant pressure (0.8 Pa) allows the deposition of more compact Cr films. The columnar structure similar to the one obtains by DCMS slowly transforms in a dense morphology without any porosity. The hardness of the Cr films increases from 9 to 16 GPa while their Young modulus remains close to the bulk value. The peak current increases with the deposition pressure while the peak voltage has the opposite trend. The films deposited by DOMS have a strong [110] preferential orientation in contrast to the films deposited by DCMS which show mixed [110] and [111] preferential orientations.

Keywords: HIPIMS, DOMS, Sputtering, Cr, Thin films

Combination of HiPIMS and PBII for the generation of high functional surfaces

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Due to the high flexibility of magnetron sputtering, based on a variety of suitable materials for deposition, the field of applications is very broad. This includes e.g. surface enhancement for optical technologies, scratch and wear resistance, anti-finger print coatings, photocatalytic coatings as well as the utilization in the semiconductor industry. The magnetron sputtering process can be operated in DC, pulsed DC or in RF-Mode.

High Power Impulse Magnetron Sputtering (HiPIMS) is a comparatively new technology and its application potential is very high. It is distinguished from conventional sputtering methods by a significantly higher ionization rate of target ions up to 80% and therefore enables a defined production of coating structures. Furthermore, HiPIMS is advantageous due to its lower thermal stress towards the treated samples and the high surface homogeneity even for complex shaped substrates. Especially, the homogenously coating of edges has to be emphasized.

To further improve the HiPIMS coating process, the combination with Plasma Based Ion Implantation (PBII) is very promising. The high density of target ions combined with their high ionization rate is a perfect basis for ion implantation of selected materials. Hence, the generation of doped coating systems even on 3D-objects is possible.

The presentation deals with results concerning the deposition of copper and silver based coating systems via PBII coupled HiPIMS. The focus of the work was to develop a doping process for coatings on polymers. To characterize the modified surface the elemental composition in combination with depth profiles, the roughness and the crystalloid structure were analyzed.



Fig. 1 Pictures of the vacuum chamber (left) and the HiPIMS discharge at the copper target (right).

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Corrosion Properties of TiN Thin Films Grown by Combined HIPIMS/DCMS and Arc Evaporation Techniques

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In this work, corrosion properties of TiN thin films grown by combined HIPIMS/DCMS and arc evaporation techniques have been studied. The effect of metal- rich core of the droplets on the corrosion resistance of TiN coating deposited by arc evaporation is discussed and the corrosion behaviour of such coating is compared to the corrosion behaviour of TiN coating grown by combined High Power Impulse Magnetron Sputtering/dc Magnetron Sputtering (HIPIMS/ DCMS), For this research, an industrial size Hauzer HTC 1000-4 system enabled with HIPIMS technology was used for the deposition of combined HIPIMS/DCMS-TiN coating. It was observed that in arc deposited coatings, when immersed in an electrolyte, a galvanic couple is formed between the droplet core (Ti-rich) and the surrounding TiN matrix, which triggers localized corrosion. To better reveal this mechanism the core of the droplets was deliberately exposed by gentle polishing of the as deposited arc coatings. The corrosion behaviour of the coatings was studied by potentiodynamic polarisation tests (ACM instruments potentiostat, -1V to +1V) using 3.5% NaCl solution. It was found that when polished, the corrosion performance of the arc-TiN coatings deteriorated significantly due to severe pitting of the exposed metal-rich core of the droplets. It was further demonstrated that the droplet free HIPIMS/DCMS-TIN coatings outperformed arc-TiN coatings. Raman analysis was used to study the constitution of the corrosion product and evaluate the corrosion damage. Scanning electron microscopy (SEM, planar view) was used to examine as deposited and corroded coating surfaces to define morphological differences.

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Effect of peak power on Cr thin films deposited by HiPIMS in DOMS (Deep Oscillations Magnetron Sputtering) mode.

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The advent of High-power Impulse Magnetron Sputtering (HiPIMS) and its variant Modulated Pulsed Power Magnetron Sputtering (MPPMS) allowed a new knob to control the flux of sputtered species, i.e., a new knob to control the kinetic effects on the growing film. Recently, a new design of the MPPMS pulses has been proposed to reduce arc generation. This form of high power pulses has been named deep oscillation magnetron sputtering (DOMS). An example of the discharge voltage and current oscillating waveforms used in this work is shown in figure 1. Each DOMS pulse consists of a packet of single oscillations. The voltage and current gradually increase to their maximum value (V_p and I_p) during the voltage on-time (t_{on}) , and then gradually decay, reaching zero before the end of the oscillations period (T).

In this work, the influence of the kinetic effects induced by a DOMS discharge on Cr sputtered thin films was studied and compared to a DCMS discharge. The Cr thin films were deposited with increasing peak power at the same average power (1.2 kW) in order to minimize changes in the thermal effects that also influence film growth. The Cr films deposited by DCMS have a columnar morphology, a [110] preferential orientation, hardness between 7.2 e 8.5 GPa and a maximum Young modulus of 255 GPa, always lower than the value of the bulk material. Although substrate polarization up to -110 V was used, some porosity always remained in the DCMS films.

The deposition rate of the Cr films deposited by DOMS decreases from 60 to 30% of the DCMS deposition rate with increasing peak power. The films also have a [110] preferential orientation. Increasing the peak power changes the film morphology from columnar to dense, increases the hardness up to 17 GPa, increases the lattice parameter and decreases the grain size. The Young modulus of the films is always close to the bulk material value showing all the films are porosity free. Bombarding the growing film with Cr ions allows a complete elimination of the porosity in the film, i.e., overcoming the shadowing effect, in opposition to substrate biasing in DCMS.



Keywords: HIPIMS, DOMS, Sputtering, Cr, Thin films

Figure 1 Typical I-V waveforms of a DOMS pulse used on this work

Analysis of Cu, Ti and Ni Plasma Generated by Inductively **Coupled Impulse Sputtering (ICIS)**

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Inductively coupled impulse sputtering (ICIS) is a new development in the field of highly ionised plasma processes. To eliminate the need for a magnetron, ICIS utilises a high power pulsed-RF coil to generate the plasma and a perpendicular electromagnetic field to the target in combination with high voltage DC pulses on the cathode. This arrangement of technologies allows the generation of high density highly ionised metal plasma of materials that are challenging with magnetron based processes such as magnetic materials.

This paper gives an overview of the properties of Cu, Ti and Ni plasmas by measuring the optical emission; energy resolved mass spectroscopy as well as the current and voltage waveforms.

The experimental setup comprises of a 13.56 MHz pulsed RF coil operating at a frequency of 500 Hz and a pulse width of 150 µs, which results in a duty cycle of 7.5 %. A pulsed DC voltage of 1900 V was applied to the cathode to attract Ar ions and initiate sputtering.

All measurements were made in two different modes. Mode 1 was at a constant pressure of 13 Pa and varied RF power settings of 1000 W - 4600 W examining the influence of power on the process. In mode 2 the effect of pressure on the process was investigated by keeping a constant RF power setting of 3000 W, while changing the pressure from 3 Pa to 27 Pa.

Optical emission spectra (OES) of Ar neutrals show a linear rise for increasing power in a log-log graph. Intensities of Ti and Ni neutrals also rise linearly with the slope in a log-log graph being twice as steep as for Ar neutrals. For Ti and Ni ions the slope of the intensity rises about three times faster than for Ar neutrals. These results are reconciled with a model which is based on electron collision as the primary excitation and ionisation mechanism for RF magnetron processes. While the Ar neutral line in the Cu plasma rises as in the Ti and Ni plasmas, the Cu neutral and ion slopes do not adhere to the model prediction.

OES intensities for increasing process pressure show a linear rise for Ar and Ni neutrals at pressures of 3-10 Pa and remain constant for pressures up to 27 Pa. Ni ion intensities remain constant for all pressures. ERMS results for Ni1+ show a non-maxwellian ion energy distribution with a high intensity peak at 20 eV and a lower intensity peak at 170 eV.

The results for pressure varied OES and ERMS of Cu and Ti plasmas are currently being analysed. Current and voltage waveforms exhibit a fast rise to the peak value with the current following the voltage. After reaching the peak values there is a steep decline to a value which is held until the end of the pulse. For higher RF powers there can be a tie-in in the voltage after the peak, before increasing to a sustainable level again. For the highest power settings a breakdown of the current and voltage can be observed.

The current work has looked at the plasma properties of Cu, Ti and Ni ICIS plasmas. These results give an overview of the plasma properties for materials with different characteristics that can be found in many more materials. Especially the results for magnetic materials are very promising for the deposition of materials that have been difficult to utilise in conventional magnetron sputtering.

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Dynamic and isothermal oxidation behaviour of HIPIMS deposited Mo-W doped carbon-based coating

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Wear resistant, low friction and thermally stable tribological coatings are in high demand for enhancing the overall tribological performance of the moving engine parts. A very essential characteristic of such coatings is to retain their stability at high temperature in both dry and lubricated conditions. The current research addressed the development and performance of molybdenum and tungsten. (Mo-W) doped carbon-based coatings in dry atmosphere at high temperature. The coatings were deposited by the combined high power impulse magnetron sputtering (HIPIMS) and unbalanced magnetron sputtering (UBM) technique on stainless steel substrate. In this paper the dynamic and isothermal oxidation behaviour of the coatings in air is reported in detail.

Thermo-gravimetric tests were carried out on three HIPIMS deposited Mo-W doped carbon-based coatings with different metal concentrations (10.5%, 17.8% and 20%), in order to study the dynamic oxidation behaviour up to 1000°C. In the same experiment, a standard state-of-the art hydrogenated DLC coating produced by plasma assisted chemical vapour deposition (PACVD) technique was used as a benchmark. The dynamic oxidation curves of all the Mo-W doped carbon-based coatings showed no change up to 400°C. A minor change in mass gain was detected in the temperature range 500°C – 700°C and then a sudden sharp rise was observed at ~800°C, indicating the onset of rapid oxidation. The hydrogenated DLC coating showed constant mass loss after 380°C due to intensive evaporation of carbon, (carbon converting to CO and CO₂) and spalation from the substrate.

Based on this dynamic oxidation behaviour, the Mo-W doped carbon-based coatings were subjected to further isothermal oxidation tests. These tests were carried out in the temperature range 400°C - 800°C depicting the initial stages of oxidation. The phase composition of the oxidised samples was examined using XRD, EDX and Raman spectroscopy techniques. The XRD and EDX analyses revealed that the coating with high metal content (20%) initiated oxidation at 400°C, whereas the other coatings showed the first evidence of oxidation at 500°C. The substrate was exposed for all the coatings when heated to 600°C. This was evidenced by the presence of oxides based on the substrate elements like Fe_xO_y and Cr_xO_y in the XRD patterns and the existence of Fe and Cr in EDX spectra. With increasing the temperature up to 800°C, the oxidation process developed further and metal oxides (Fe₂O₂ and Cr₂O₂) and carbides (Cr_xC_y) were formed. It is believed that the metal carbides were formed by a combination of out-diffusion of chromium into the coating and diffusion of carbon into the substrate.

Observations from Raman analysis further consolidated the XRD and EDX results. For the coating with lower metal content (10.5%) the phonon bands for disordered and ordered graphite (D and G) were observed up to 700°C indicating availability of free carbon in the coating. However, at high temperature the ID/IG ratio was reduced, which indicates change in the bonding structure of the graphitic phase. For the higher metal content coatings (17.8% and 20%) the D and G bands were found to be present up to 500°C, however they disappeared with further increase in temperature (600°- 800°C). The absence of graphitic carbon bands at these higher temperatures indicates that the coatings were predominantly transformed of metal oxides phases such as W_xOy, MoO_x, Fe_xO_y and Cr_xO_y. These studies showed that HIPIMS deposited Mo-W doped carbon-based coatings show relatively high thermal stability (first evidence of oxidation at 500°C), whereas the PACVD deposited hydrogenated DLC coating deteriorate their protection properties after 380°C.

Abstract Book

Development of HIPIMS technology for superconductive coated cavities

HIPIMS is gaining large consensus around the world as a possible solution to overcome the problems faced with standard dcMS for the superconductive thin film coatings on copper RF cavities. Given the wide parameter space available with HIPIMS it is informative to draw out th relationship between plasma parameters microstructure and guality of the film produced. Influence of different discharge settings (pulse width, current density and frequency) has been studied in order to improve film performance. Samples have been produced in order to analyse the film microstructure, correlated to the plasma parameters, as well as superconductive properties. The microstructure showed an interesting behaviour, with the grain size increasing with the peak discharge current; the Residual Resistance Ratio (RRR) is inversely proportional to the current for short pulse widths, while it is directly proportional to the current for longer pulse widths. This seems to be related to an increasing number of grains with (110) crystallographic orientation in the deposited film. The performance of superconductive cavities produced with HIPIMS is comparable with some of the best dcMS coated ones.

Interesting results are obtained with OES and MS comparing argon and krypton process gases. In particular more energetic ions are produced when using krypton as process gas due to the longer mean free path for elastic collisions for the same pressure. The effect of the biasing voltage on the film structure has also been investigated. Experiments on cavities have been conducted at CERN while samples have been prepared both at Sheffield Hallam University and at CERN. This allows us to make a comparison between the two different experimental setups. Results on plasma analysis, superconductive properties and film morphology will be presented as well as the performance of the latest HIPIMS-coated cavities.

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Different process parameters controlling reactive high-power impulse magnetron sputtering of dielectric oxide films

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High-power impulse magnetron sputtering (HiPIMS) with a pulsed reactive gas (oxygen) flow control [1] was used for highrate reactive depositions of densified stoichiometric ZrO2, HfO2 and Ta2O5 films on a floating substrate. The depositions were performed using a strongly unbalanced magnetron with a planar Zr, Hf and Ta target of 100 mm diameter in argonoxygen gas mixtures at the total pressure close to 2 Pa. The repetition frequency was 500 Hz at the average target power density from 29 Wcm-2 to 54 Wcm-2 during a deposition with duty cycles from 2.5 % to 10 %. The target-to-substrate distance was 100 mm. For the same duty cycle of 10 %, the deposition rates were up to 120 nm/min for the ZrO2 films, up to 125 nm/min for the Ta2O5 films and even up to 345 nm/min for the HfO2 films. In this presentation, we report on discharge characteristics during the controlled reactive HiPIMS of these films and on important quantities characterizing the reactive sputter deposition processes, namely, the deposition rate of films, the deposition-averaged oxygen flow rate and the deposition rate per deposition-averaged oxygen flow rate. A particular attention will be paid to the choice of a suitable process control parameter, either the average discharge current in a period of a pulsed power supply or the oxygen partial pressure in a vacuum chamber, for a given deposition. It is based on a time-dependent response of these two process parameters to constant flow rate pulses of oxygen into the chamber.

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Oral session preferentially.

Ag- and Cu-flexible surfaces leading to fast bacterial inactivation in the dark: comparative study DCMS vs **HIPIMS** depositions

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Abstract

Compact uniform and adhesive films of Ag and Cu on flexible substrate have been prepared by DC-magnetron sputtering (DC) and high power impulse magnetron sputtering (HIPIMS). This study reports the HIPIMS deposition for Ag and Cu on textile fabrics, the bacterial inactivation kinetics and the nature of the species in the plasma produced during HIPIMS sputtering. The deposition rates of Ag and Cu atoms and the bacterial inactivation times in the dark reported as a function of the peak current during the sputtering processes. The atomic percentage concentrations changes of the surface elements and the changes in the oxidation states of Ag and Cu were monitored during E. coli inactivation by X-ray photoelectron spectroscopy (XPS),. The Ar and metal-ions produced in the magnetron chamber were determined quantitative mass spectroscopy (QMS). A mechanism is suggested for the bacterial inactivation in the dark for the Ag and Cu sputtered surfaces.

Si_vN_x coatings deposited by reactive High Power Impulse **Magnetron Sputtering**

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Abstract

Reactive high power impulse magnetron sputter (HiPIMS) processes for the deposition of Si_vN_x films were investigated with respect to Si, N, properties as well as the plasma composition, particle energy, and discharge characteristics, Additionally, experimental results are complemented by first-principles calculations based on density functional theory. These calculations on most abundant plasma precursor fragments provide information on their relative stability, abundance, and reactivity. The Si_vN_x film growth and plasma characterization were conducted in an industrial coater, using pure Si targets and N2 as reactive gas. The impact of different process parameters such as deposition pressure, N2-to-Ar flow ratios varied between 0 and 0.31, pulse frequencies ranging from 100 to 1000 Hz as well as different average power settings on the Si_vN_x deposition process and the film properties were investigated. Furthermore, the metallic and reactive HiPIMS processes were studied with time resolved and time averaged positive ion mass spectrometry, using the most abundant Si and N isotopes as well as precursor fragments to characterize the plasma. Mass spectrometry results show high relative amounts of ionized isotopes for N₂-to-Ar flow ratios lower than 0.16 during the initial part of the pulse, while relative signals from ionized molecules rise with the N₂-to-Ar flow ratio at the pulse end. The films were characterized with respect to their composition, chemical bonding and residual stresses as well as mechanical properties by X-ray photoelectron spectroscopy, elastic recoil detection analysis, X-ray diffraction, and nanoindentation, respectively.

The Si, N_x deposition processes and film properties were found to be influenced primarily by the N₂-to-Ar flow ratio and thus the nitrogen content in the Si_vN_v films as well as to a lower extent by the HiPIMS frequencies and power settings. At increased N₂-to-Ar flow ratios the deposition rates, the amount of incorporated Ar, adsorbed surface O and residual stresses decrease, whereas the hardness and the H/E ratio increased.

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Effects of HIPIMS Deposition Pressure on (Ti, AI)N Film Properties at Inner Wall of Sub-Millimeter Scale on Small Holes

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Abstract

Conventional hard film deposition process confronts the difficulties in the deposition of high performance films on the inner wall of closed shaped micro-dies with sub millimeter scale holes. In order to enhance the tool life as in industrial scale, thin films with anti-adhesive and high wear toughness are required to deposit uniformly on the three dimensional complicated shape structure. Focusing on the great possibility of HIPIMS deposition, the authors demonstrated its availability and practical advantages for the microforming die in the previous report. For the further enhancement of the thin film properties at inner wall of sub millimeter scale small holes, the present study investigated the impact deposition pressure during the HIPIMS process. As representative of the basic nitrides thin files (Ti, AN)N films was deposited by HIPIMS under various pressures of 0.5, 1, 2 Pa on the sub millimeter scale small holes. This small hole structure was realized by clamping the comb shaped stainless steel plates with two flat high speed steel (HSS) substrates. To characterize the film properties at different position of inner wall depth, the several analytical techniques were performed. A field emission scanning electron microscopy (FE-SEM) was used to evaluate the surface morphology and cross sectional microstructure, which is prepared by a focused ion beam process (FIB). Additionally, the local elementary composition at each position of inner wall depth was analysed by an energy dispersive X-ray spectroscopy (EDX) coupled with the above FE-SEM. As results, the higher deposition rate at the inner wall of tiny holes was shown for the lower pressure conditions as expected. Furthermore, the local chemical composition analysis showed the varying tendency from the entrance to the deeper position of the holes. Particularly, the deposition pressure affects strongly the elementary composition ratio of Ti / Al and N / (Ti, Al) at each position of the inner wall depth. Based on these results, the (Ti Al)N thin film growth at sub millimeter scale holes and the appropriate HIPIMS deposition pressure conditions to obtain the uniform and high wear-resisted film coating were discussed in view of the application to microforming die.

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HIPIMS deposition of p-type iridium oxide films

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Abstract

One of the important requirements for today's electronic applications, like displays, electro-chromic devices, light emitting diodes or solar cells, is a stable and if necessary a transparent p-type conducting film. Iridium oxide (IrO_2) has been studied for its p-type conductivity for many years using DC and RF sputtering technologies. In this current work we deposited IrO_2 using high power impulse magnetron sputtering (HIPIMS) at room temperature. For these depositions various process parameters like oxygen flow, total pressure, pulse frequency, duty cycle and power applied at the target as well as target to substrate distance were varied and studied. Further, we also deposited IrO_2 with RF sputtering in order to compare them with the HIPIMS films.

The resulting electrical, optical and morphological characteristics of these films were analyzed for their p-type conductivity and transparency. The changes in the film properties with the changes in process variables and also the results of these analyses will be discussed and presented. The optimized IrO₂ film was tested for its p-type conductivity by replacing the p-doped layer in a pin-amorphous Si:H solar cell.

Keywords: HIPIMS, p-type oxide, iridium oxide

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Investigation of ionized metal flux in enhanced High Powe Impulse Magnetron Sputtering discharge

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High Power Impulse Magnetron Sputtering (HiPIMS) is the term for dc-pulse magnetron discharge operated at low repetition frequency (typically about 100 Hz) with duty cycles of about 1%. This magnitude of duty cycle together with applied average current density $/_{av} \approx 20-40$ mA/cm² to the magnetron cathode results in a high current density in pulse 2 - 4 A/cm² and production of high density plasma with electron and ion concentration $n_{e,i} \approx 10^{18}-10^{19}$ m⁻³ where high fraction of sputtered particles is ionized. Ionized species to be deposited are easily affected by an electric field and their energy and direction can be manipulated. Further, it was shown that film properties (smoothness, density, adhesion, crystallography, etc.) improve if sputtered particles are ionized. Our attention is focused on the measurement of ionized flux and degree of ionization of sputtered species in assisted ECWR-HiPIMS discharge.

Measurements were performed in HiPIMS discharge supported by Electron Cyclotron Wave Resonance (ECWR) plasma. Continuously driven ECWR discharge provides a pre-ionization effect which allows (i) a significant reduction of the working pressure, (ii) results in an enhanced ionization of metal atoms and (iii) leads to a faster ignition and development of the HiPIMS pulse. The magnetron cathode was equipped (stepwise) with targets made from different materials (Ti, Cu, Al, Fe).

The ionization of sputtered species was measured in wide range of pressures using modified quartz crystal microbalance (QCM) biased with a positive voltage V_b applied on the collecting electrode coated on the quartz crystal. The modification of QCM device is based on a new technical configuration capable to separate electrons from the surface of quartz crystal collecting electrode when the positive bias V_b is applied for repelling of all positively ionized particles. No electrostatic grids are necessary in this configuration. All deposited species (ions, neutrals) contribute to the total flux when V_b potential is lower than the plasma potential V_{pl} . In contrast, the positive ions are repelled when $V_b > V_{pl}$ and only neutral species yields to the deposition rate. In this way, it is possible to get information about ratio of ionized and neutral fluxes of depositing particles incorporated in the deposited film.

Measurement carried out by energy-resolved mass spectrometer was used for qualification of ionized flux. It was found that ionization degree of sputtered particles depends on pressure significantly. Ionized metal species M⁺, M²⁺ and neutral M are typically observed at low pressures while M⁺ become dominant if pressure increases.

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Simulation of Neutral Particle Transport in High Power Impulse Magnetron Sputtering

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The spatial and temporal evolution of plasmas in the high power impulse magnetron sputtering (HiPIMS) regime as well as the dynamics of the neutral gas species are crucial for the understanding of the overall plasma deposition process. The plasma is locally highly ionized and as such the neutral gas is no longer a constant background in these ionization zones. Moreover, the interaction of energetic sputtered particles with the neutral gas can result in substantial gas heating and therefore strong rarefaction. From a modeling perspective, due to the low operating pressures typically below 1 Pa, a kinetic description of the involved gas dynamic and plasma processes is required [1]. In this work, the modeling of the described processes is approached in terms of a modified version of the threedimensional Direct Simulation Monte Carlo (DSMC) code dsmcFoam [2], which is part of the open source CFD toolbox OpenFOAM [3]. A time-varving flux of sputtered aluminum particles is imposed at the target of a generic cylindrical plasma reactor geometry. The kinetic simulation of the former mimics the strong sputtering wind resulting from a short current pulse typical for HiPIMS discharges. This allows us to investigate the transport of sputtered aluminum particles through a neutral gas atmosphere of argon and its impact on the latter. First, we analyze the particle densities and fluxes inside the described discharge reactor. We next investigate the influence of different pulse parameters, e.g., the pulse duration, the repetition rate, or the sputtered particle flux of current pulse on the individual particle densities and fluxes. We comment on the significance of the mentioned discharge parameters in particular with respect to the inherent time scales and time delays respectively. Finally, we discuss the observed spatiotemporal evolution and determine the significance of the sputtering wind for the dynamics of the neutral argon gas. As expected, we find its influence to be largest in the region below the mimicked race track where the strongly ionized plasma zones are assumed.

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Influence of pulse on time and frequency variation on the properties of Cu coatings performed by HiPIMS power source

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Abstract:

HiPIMS is the novel technology used for physical vapour deposition where high peak power unipolar pulses are applied to the target. HiPIMS offers benefits of high plasma density, better adhesion of coated films, high coating density and very low surface roughness. In addition to this, it is possible to dump peak power up to few MW without unnecessary increase of cathode temperature. Characteristic for HIPIMS is that the power is supplied to the sputter target in short pulses of few us at low frequencies. As a result high density plasma is produced leading to a large increase of the ionization probability of the sputtered material. The high ion-to-neutral ratio in HIPIMS enables the deposition of dense and smooth metallic and compound films. The present investigation focused to study the influence of the pulse parameters on the surface properties of the Cu films. The results are observed by varying the frequency and pulse on time between 10 -100Hz and 10uS - 200uS respectively. The coated films are characterised by SEM, XRD and AFM. It has been observed that the decrease of pulse length at constant mean power gives considerable increase of cathode current and the deposition rate reduced with decreasing pulse on time. Also HiPIMS deposited coatings were approximately 5-15% denser compared to the DC sputtering.

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Benefits of the controlled reactive high-power impulse magnetron sputtering of dielectric films

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We have proposed a pulsed reactive gas flow control (RGFC) of the reactive high-power impulse magnetron sputtering (HiPIMS) to avoid substantial problems with arcing on target surfaces during reactive sputter depositions of dielectric films at high target power densities and with low deposition rates achieved [1].

Using this process control, we are able to maintain sputter deposition of the dielectric stoichiometric films in the region between a more and less metallic mode, and to utilize exclusive benefits of the HiPIMS discharges, such as intense sputtering of atoms from the target, very high degrees of dissociation of RG molecules in the flux onto the substrate, strong "sputtering wind" of the sputtered atoms, highly ionized fluxes of particles to substrate and enhanced energies of the ions bombarding the growing films, in preparation of the films.

HiPIMS with the pulsed RGFC was used for high-rate reactive depositions of densified, optically transparent zirconium and hafnium dioxide films on a floating substrate at a distance of 100 mm from the target. An optimized location of the oxygen gas inlets in front of the target and their orientation to the substrate surface made it possible to improve quality of the films due to minimized arcing at the sputtered target and to enhance their deposition rates up to 120 nm/min for the zirconium dioxide films and even up to 345 nm/min for the hafnium dioxide films at a deposition-averaged target power density close to 50 Wcm⁻² and a voltage pulse duration of 200 µs. Details of the deposition processes, including an energy-resolved mass spectrometry at the substrate position and computer simulations, and measured properties of the films will be presented.

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Structural, electrical and optical properties of Zn-Ir-O thin films

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Transparent conducting oxides (TCOs) are the metal oxides having good optical transparency and high electrical conductivity. The present potential applications of TCO thin films are transparent electrodes for at panel displays and photovoltaic cells, low emissivity windows, window defrosters, transparent thin films transistors, LEDs and semiconductor lasers [1]. One of the obstacles to further developments of transparent electronics based on transparent conductive oxide thin films is lack of p-type conductors.

Amorphous ZnO-IrO2 thin films deposited by pulsed laser deposition at room temperature are reported to be potential p-type TCOs [2]. In the present study, we have investigated properties of Zn-Ir-O thin films deposited by reactive DC magnetron sputtering.

Zn-Ir-O thin films were deposited on glass, Si and Ti substrates by reactive DC magnetron sputtering from a metallic Zn (99.95 %) target with Ir pieces on the target surface in an Ar+O2 atmosphere. A set of samples was deposited at different oxygen to argon gas ratios (1/4, 1/2/, 1/1) and at different fractions of iridium on the zinc target erosion zone (1, 3, 5, 7, 10, 13, 15 %). Composition and structural, electrical and optical properties of the Zn-Ir-O thin films were studied by XRF, XRD, Raman, FTIR techniques, as well as Hall effect measurements and two beam optical spectrophotometry.

Preferential sputtering of iridium atoms compared to zinc was observed. As-deposited Zn-Ir-O thin films at room temperature are x-ray amorphous. Increase in iridium concentration leads to lower resistivity, but higher absorption coeffcient in the visible range [3].

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