BOOK OF ABSTRACTS

2nd International Conference on Fundamentals and Applications of HIPIMS

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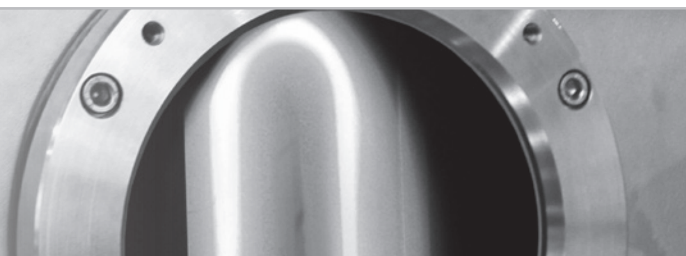
HIPIMS 2011

2ND INTERNATIONAL CONFERENCE ON HIPIMS



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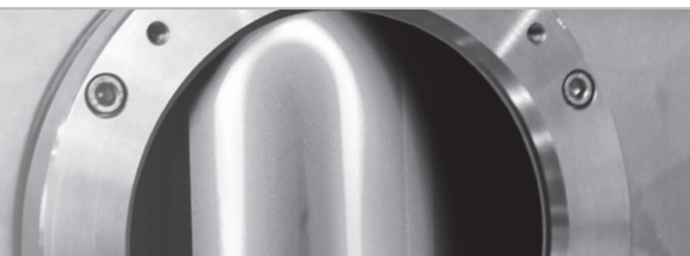

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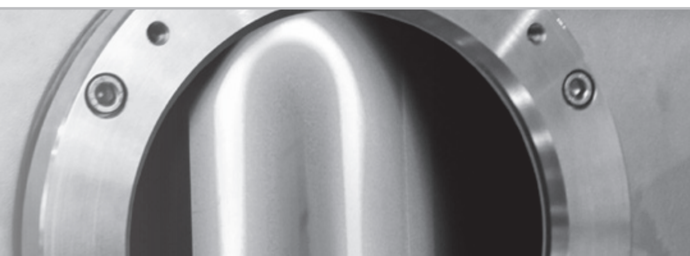

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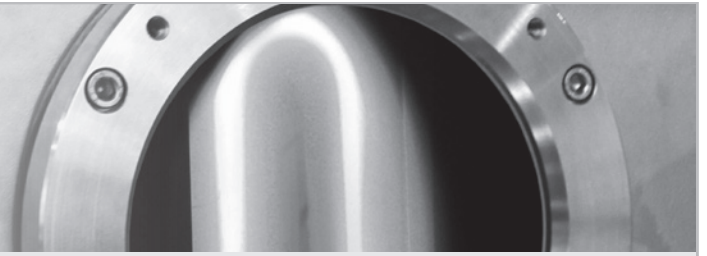


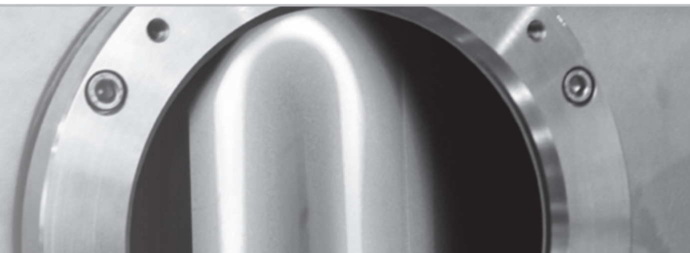
HIPIMS coatings: a status report on the introduction of industrial coating applications.

Thomas Krug, Roel Tietema*, Frank Papa, Ivan Kolev, Paul Peeters

Hauzer Techno Coating BV
Van Heemskerckweg 22
5928LL Venlo
Netherlands

***Corresponding Author**
Roel Tietema

HIPIMS coatings have been under development since the end of the nineties. In this presentation an overview of the developments from the perspective of Hauzer will be given, beginning from the first discussions on participation in a CRAFT project, then continuing from the actual first steps made by Hauzer in the field of HIPIMS and finally leading to the hardware and process developments at Hauzer. The cooperation with SHU has been an enabling factor in the successes accomplished by Hauzer today.
Hauzer has reached a good understanding of the process controls required to produce high performance coatings. The technology that has been applied will be addressed. The final development of processes has lead to excellent results. These independent results will be shown in the presentation. Besides the technological benefits, the commercial feasibility is of course of importance. Commercial aspects will be reviewed in this talk, since from industrial perspective this is an aspect of high importance that is playing a major role for a success in a broad future expansion of the technology.



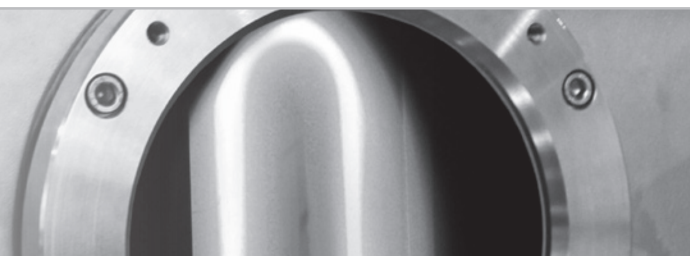
HIPIMS goes to production

O. Lemmer, C. Schiffers, S. Bolz, W. Kölker

CemeCon AG
Adenauerstr. 20 A4
D-52146 Würselen
Phone: 02405-44-70-410
E-Mail: Oliver.Lemmer@CemeCon.de

HIPIMS technology has experienced a rapid development during the last few years. Starting with first industrial HIPIMS power supply equipment only a few years ago and a lot of institutional research during the following years HIPIMS has now reached a point where market appearance is a must. The predicted technological benefits with respect to plasma ionization and coating design can now be exploited for industrial products. Whereas some working groups have focused on the use of HIPIMS for ion etching improvements the most important implications can be derived for the coatings themselves.

Therefore HIPIMS has gained an unlimited importance for the whole PVD process. Properties like coating morphology, density, oxidation resistance, adhesion on the substrate, hardness, Youngs modulus and toughness can be tuned for better application performance. Tool manufacturers and industrial users have now began to use this novel technology in practice. The presentation gives a current status report on tool performance and on the integration of HIPIMS-technology into the industrial practice. Future needs as a guidance for the HIPIMS-community are addressed.



Domino Platform: PVD Coaters for Arc Evaporation and High Current Magnetron Sputtering (HIPAC)

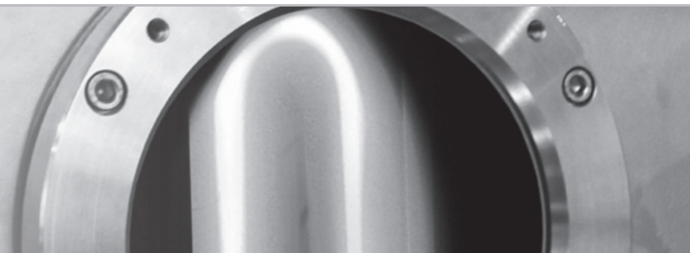
J. Vetter*, J. Müller, T. Rasa, G. Erkens

Sulzer Metaplas, Am Böttcherberg 30-38,
51429 Bergisch Gladbach
www.sulzer.com

***Corresponding Author**
joerg.vetter@sulzer.com

Two PVD deposition methods are applied by Sulzer Metaplas to produce tribological and functional coatings: the vacuum arc evaporation and the magnetron sputtering. The application of vacuum arc evaporation is the dominating PVD method for tool coatings and high performance component coatings. Besides advanced hard coatings (MAC's) also upgraded evaporators like APA (advanced plasma assisted) are available. Classical DC-magnetron (DC-MS) sputtering is mainly used for a-C:H:Me-coatings, or for the deposition of interlayers for a-C.H:X coatings.

Although the DC-magnetron sputtering process often results in smoother coatings (no or low droplet formation by micro arcing) than the arc evaporation the number of coating systems in industrial use is much lower than that of the arc evaporation. The reasons for that are the advantages (stability, productivity, costs, coating properties) of the arc evaporation process in industrial use. High current pulsed magnetron sputtering (HCP-MS) processes (trade names HIPIMS, HPPMS, MPP, HIPAC and others) were developed over the past two decades starting with the finding of the high ionization level of the self-sustained magnetron sputtering. This higher ionization level opens up new possibilities of the magnetron sputtering using targets with sufficient electrical conductivity. A demand of R&D and small volume production systems is obvious. One solution provided by Sulzer Metaplas is the DOMINO mini system. The DOMINO platform will be described both as an arc evaporation system and as a HCP-MS system. Selected results of both processes like coating properties and deposition rates will be presented.



New features in HUETTINGER's HIPIMS power supplies

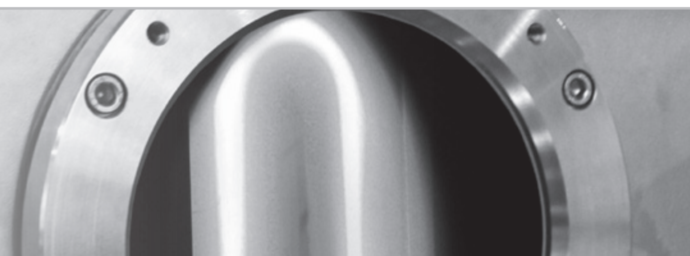
W. Glazek*, A. Klimczak, P. Ozimek, P. Różański

HUETTINGER Electronic Sp. z o.o., Marecka 47,
05-220 Zielonka, Poland

***corresponding Author**
Wojciech.Glazek@pl.huettinger.com

HUETTINGER is the first company offering industrial power supply enabling users to sputter materials with HIPIMS technology. At the moment HUETTINGER is looking for ways to support growing demand for new features in HIPIMS need in order to progress with the technology, and to support developing industrial applications.
One way to support the HIPIMS technology is to enhance properties of the power supply when it comes to internal control system. Making it faster and more precise gives user enhanced control over the process, and enables power supply to react to process parameters faster.

Other way is to develop new features and solutions. These new features would support especially areas where there is significant potential for the technology to grow. One of such areas is reactive sputtering with HIPIMS.
New features supporting reactive sputtering with HIPIMS technology were implemented into TruPlasma Highpulse Series 4000 HUETTINGER's power supply. The solution enabled to deposit coatings with good quality and at high deposition rate. Results of use of the solution will be presented and potential for industrial applications will be discussed.



Overcoming the geometrical limitations of conventional sputtering by controlling the ion-to-neutral ratio during HIPIMS

G. Greczynski, J. Jensen and L. Hultman
Department of Physics, Linköping University,
583 31 Linköping, Sweden
phone: +46 13 28 1213 | E-Mail: grzgr@ifm.liu.se

High power pulsed magnetron sputtering (HIPIMS) has been used to grow thin chromium films on substrates facing and orthogonal to a rectangular shaped target. It is demonstrated that at low peak target current density, $jT < 0.6$ A/cm^2 corresponding to a low ion-to-neutral flux ratio, films grown on substrates facing the target exhibit substantial in-plane alignment. This is caused by the asymmetry in the off-normal flux of sputtered species. With increasing jT the biaxial alignment degrades, as the major portion of the incoming flux (metal ions) can be effectively steered by the electric field of the substrate to eliminate asymmetry imposed by geometrical restrictions. Eventually, at $jT = 1.7$ A/cm^2 a fiber texture is obtained. For films grown on substrates orthogonal to the target (cf. cross sectional SEM micrographs in Fig. 1), the large column tilt characteristic for growth by conventional DC magnetron sputtering (Fig. 1a) and also observed for films prepared by HIPIMS at low jT (Fig. 1b), decreases with increasing ion content in the flux and almost disappears at the highest value of jT (Fig. 1e). The latter deposition condition corresponds to a high ionization of the material flux to the substrate 1 so that deposition takes place along substrate normal despite the high nominal inclination angle. Thus, in the limit of high jT the artifacts of conventional PVD, resulting from the line-of-sight deposition, are eliminated and the film growth proceeds more or less unaffected by the substrate orientation. Samples mounted orthogonally thus possess a similar texture, morphology, and topography as those facing the target.

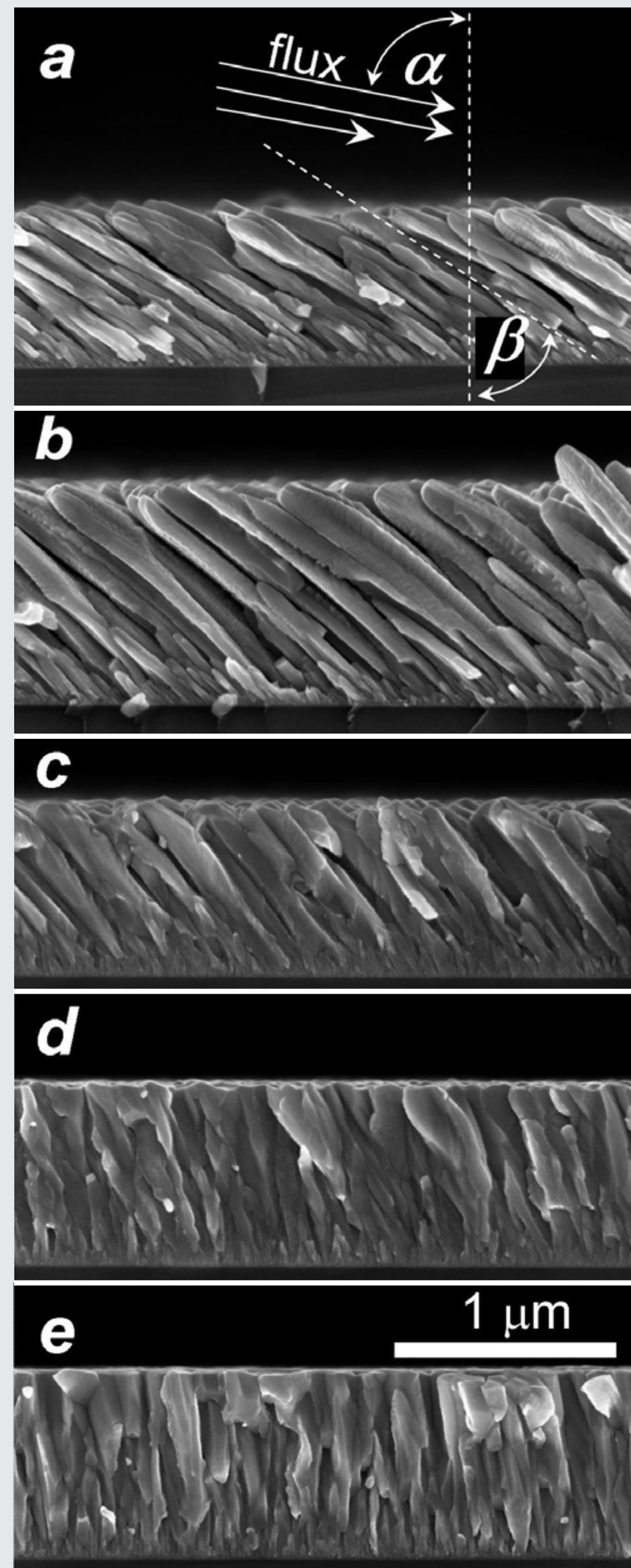


Fig. 1 Cross sectional SEM micrographs of films deposited on substrates oriented orthogonally to the target: a) DCMS reference and b) – e) HIPIMS samples with increasing peak target current density b) $jT = 0.1$ A/cm^2, c) $jT = 0.6$ A/cm^2, d) $jT = 0.9$ A/cm^2, e) $jT = 1.7$ A/cm^2. The scale is the same for all micrographs.

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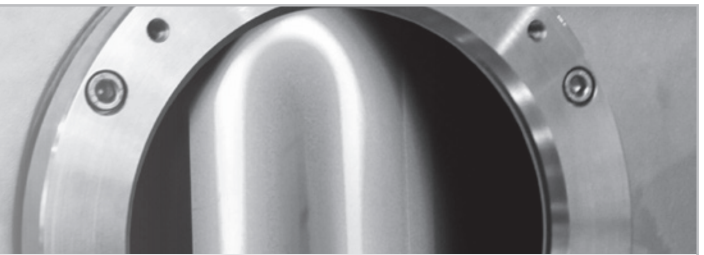


Modelling of target effects in reactive HIPIMS

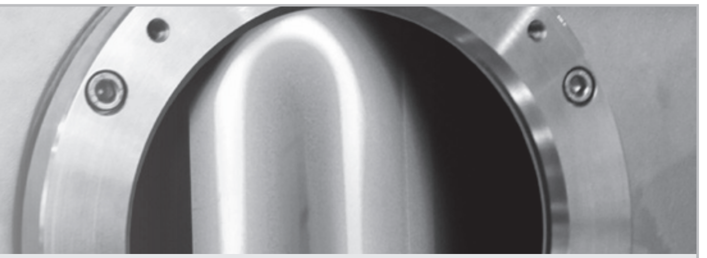
T. Kubart*, J. Andersson

Solid State Electronics, The Angstrom laboratory, Uppsala university, Box 534, 751 21 Uppsala, Sweden

*corresponding author
Tomas.Kubart@angstrom.uu.se
phone: +46 18 471 72 57 | Fax: +46 18 55 50 95

In reactive High Power Impulse Magnetron Sputtering of oxides, target effects such as reduced surface oxidation during off time, increased implantation of reactive gas due to the higher discharge voltage as compared to normal DC sputtering, and enhanced target cleaning during on time are considered to be responsible for the differences compared to reactive DC sputtering. These effects are assumed to cause shifts in the target oxidation state and hence lead to the hysteresis behaviour changes reported in experimental studies.

In this contribution, we have simulated the target processes using the binary collision approximation code TRIDYN. TRIDYN is a dynamic version of the popular TRIM/SRIM package and thus suitable for description of pulsed processes. The model uses substantial simplifications but takes into account changes in target surface composition, possibility of metal back-attraction and the difference between DC and HIPIMS discharge voltage. A detailed description of the model and its assumptions is provided. Using an Al target sputtered in Ar+O_2 mixture as a model system, a range of pulse configurations is simulated for different oxygen partial pressures. The results are shown and discussed in detail, with special focus on the differences from a DC sputtering process. Our results indicate that the target effects alone are not sufficient to explain the observed shift of hysteresis and its frequency dependence. Explanations relating to dynamic phenomena in the gas phase, such as gas rarefaction, are considered.



A non-stationary model for high power impulse magnetron sputtering discharges

T. Kozak, A. D. Pajdarova

Department of Physics, University of West Bohemia,
Univerzitni 22, 306 14 Plzen, Czech Republic
E-Mail: kozakt@kfy.zcu.cz

We present a non-stationary model of a high power impulse magnetron sputtering discharge. The model splits the magnetron discharge into two zones, namely the high density plasma ring above the target racetrack including a target sheath, and the bulk plasma region between the plasma ring and the substrate. By solving the particle and energy conservation equations for these two zones, the model makes it possible to evaluate time evolutions of the averaged process gas and target material neutral and ion densities, as well as the fluxes of these particles to the target and substrate during a pulse period. Consequently, the target and substrate current density waveforms, together with the effective electron temperature, can be calculated. In addition, the important deposition characteristics, such as the deposition rate, the fraction of target material ions in the total ion flux to the substrate and the ionized fraction of target material atoms in the flux to the substrate can be evaluated. The geometric input parameters of the model are the vacuum chamber dimensions, the target-to-substrate distance, the target and substrate diameters and the plasma ring size (defined roughly by the geometry of the magnetic field). The main process input parameters are the process gas pressure, the magnetic field strength, the target voltage waveform during a pulse period and the repetition frequency of the pulses. Furthermore, additional material parameters, such as the sputtering yields, the secondary electron emission coefficients, the ionization and excitation cross-sections for the process gas and target material must be provided. The effects of various process parameters on the discharge and deposition characteristics were examined. The model predictions were compared with the experimental results obtained for two different high power impulse magnetron sputtering systems. 1,2 It was shown that the model provides a good qualitative picture of the complicated processes determining the sputtering and deposition mechanisms in the high power impulse magnetron sputtering discharges investigated.

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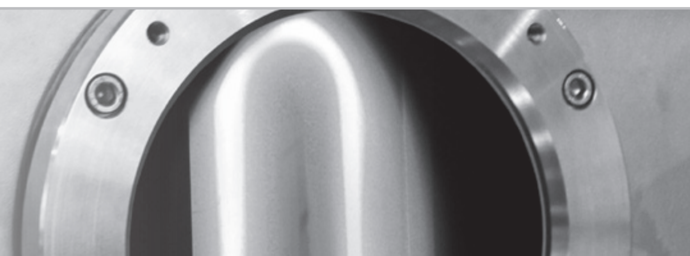

Measurement of the Energy Flux at the substrate during the growth of TiO_2 thin films by DC and High-Power Impulse Magnetron Sputtering

A. Balhamri[1, 2], P. A. Cormier[3], A. L. Thomann[3], R. Snyders[1,4], S. Konstantinidis[1]

[1] Chimie des Interactions Plasma Surface (ChIPS), CIRMAP, Université de Mons, Place du Parc 20, B-7000 Mons, Belgium

[2] Laboratoire Rayonnement & Matière: Equipe d'Optique Appliquée et Transfert d'Energie (LOPATE), Université Hassan 1er, Faculté des Sciences et Techniques de Settat, B.P. 461 Settat, Morocco

[3] Groupe de Recherches sur l'Energétique des Milieux Ionisés UMR6606, Université d'Orléans, BP6744, 45067 Orléans cedex 2, France

[4] Materia Nova Research Center, 1 Avenue Copernic, B-7000 Mons, Belgium

The energy flux transfer between the plasma and the substrate surface is of particular interest during magnetron sputter deposition of thin films, as it is a key parameter that influences phase formation and morphology of sputtered films. In this study the total energy flux at the substrate is measured directly with a heat flux sensor[1] located at the substrate position (8 cm from the target) during reactive sputter deposition of TiO_2. Deposition was performed on silicon wafers using a 10 cm in diameter circular titanium target in a reactive atmosphere (0 – 65 % of O_2 in argon at a pressure of 0.66 Pa). Magnetron Sputtering (dcMS) and High Power Impulse Magnetron Sputtering (HIPIMS)[2] experiments were carried out using unbalanced and balanced magnetron targets with an average power being set either to 400 or 800 W. The film growth rate was measured by profilometry and the amount of material deposited was obtained by X-Ray Fluorescence (XRF) and Rutherford Backscattering Spectrometry (RBS). The phase constitution of the films was investigated by X-ray Diffraction (XRD). For 65 % of oxygen (poisoned regime) at 800 W, using an unbalanced magnetron, the growth rates are 12 nm/min and 3 nm/min during dcMS and HIPIMS, respectively, the RBS measurement give us a value of 8E17 atm/cm^2 for dcMS and 6E17 atm/cm^2 for HIPIMS. The energy is 496 mW/cm^2 in dcMS and increases up to 861 mW/cm^2 during HIPIMS. From our results it appears that the energy per atom (ev/atom) deposited is dramatically increased during HIPIMS. Increasing the energy supplied to the substrate by using HIPIMS allows e.g. densification of the TiO_2 films.

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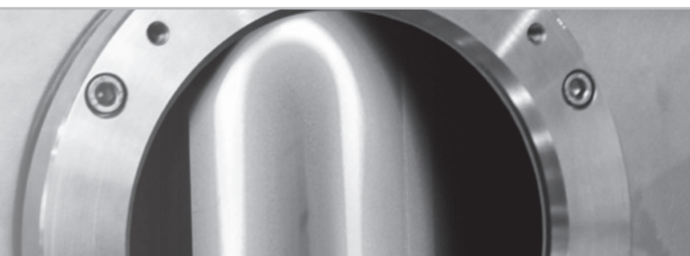


CuInSe Thin Film Photovoltaic Absorber Layers by HIPIMS

A. P. Ehiasarian[2]*, V. Sittinger[1]

[1] Fraunhofer Institute for Surface Engineering and Thin Films IST, Bienroder Weg 54E, 38108 Braunschweig
phone: +49 531 2155-602 | fax +49 531 2155-900

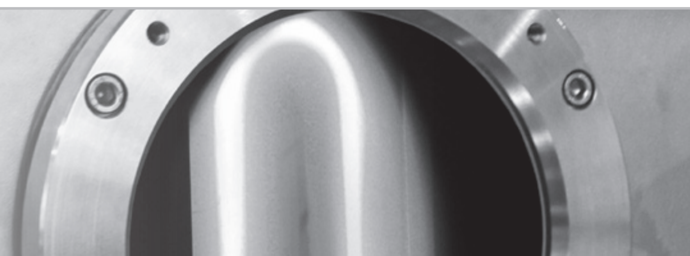
[2] Materials and Engineering Research Institute, Sheffield Hallam University, Howard St., Sheffield, S1 1WB, United Kingdom
Phone: +44 114 2253646 | Fax +44 114 2253500

***Corresponding Author**
a.ehiasarian@shu.ac.uk

CuInSe2-based thin film photovoltaics are gaining popularity in industry due to the high conversion efficiency, high productivity of the deposition process and competitive pricing against Si-based processes.

CuInSe deposition experiments were carried out in a UHV system enabled with HIPIMS technology. Energy-resolved mass spectroscopy measurements showed that the deposition plasma contained significant quantities of Cu^{1+} and In^{1+} ions which were on par with those of Ar^{1+}, whereas the Se ion flux constituted 1 % of the total. 90 % of all Se ions were atomic, but molecules of Se2, Se_3, and Se_4 were also detected within the range of the spectrometer; heavier molecules contributed consecutively less flux. The temperature of Ar^{1+} ions was 7 eV in the on-time and 2 eV in the off-time with maximum energy of 20 eV. CuInSe films were deposited by HIPIMS with different composition. Films with high Cu content $Cu_2In_{0.6}Se$, had a rough microstructure, each column comprising several facets. XRD showed low intensity broad $CuInSe_2$ (112) peak. Raman spectroscopy showed high amounts of a secondary phase.

Films with Cu-In ratio closer to stoichiometric $Cu_{0.8}InSe$ had smooth column tops. $CuInSe_2$ phase was detected by Raman and XRD and a high ratio of (112):(220) diffraction peaks was measured. No Cu_2Se was found by Raman with excitation wavelength of 532 nm. Regardless of Cu content, columns were well defined, ranging in size from 200 to 700 nm at film thickness of 1.5 µm.



Deposition rate and energy flux differences for MPP and HIPIMS pulses for Chromium, Chromium Nitride, and Aluminium

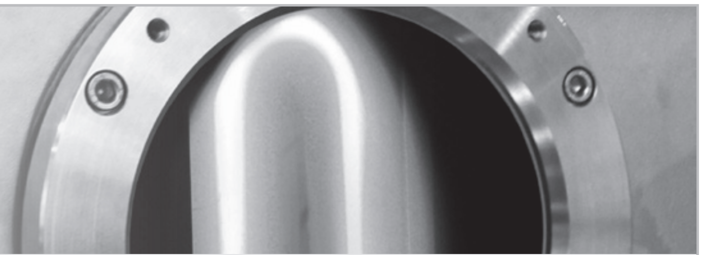
F. Papa[1], H. Gerdes[2], R. Bandorf[2], A. P. Ehiasarian[3], A. Campiche[1], A. Hendriks[1], R. Tietema[1], T. Krug[1], G. Bräuer[2]

[1] Hauzer Techno Coating BV, Van Heemskerckweg 22, 5928LL, Venlo, The Netherlands
phone: +31 77 3559781 | Fax +31 77 3559798
E-Mail: fpapa@hauzer.nl

[2] Fraunhofer Institute for Surface Engineering and Thin Films IST, Bienroder Weg 54E, 38108 Braunschweig
phone: +49 531 2155-602 | fax +49 531 2155-900

[3] Materials and Engineering Research Institute, Sheffield Hallam University, Howard St.
Sheffield, S1 1WB, United Kingdom
Phone: +44 114 2253646 | Fax +44 114 2253500

For peak current densities below 1 A/cm^2, it has been found that the pulse length and form have a strong influence deposition rate, degree of metal ionization and thus the ion/neutral ratio at the position of the substrate. Magnetic field strength also has a strong influence on these parameters. MPP and HIPIMS pulses have been compared for Cr, CrN and Al. Deposition rates can be either higher or lower for either technology depending on system configuration and the pulse characteristics. The ion/neutral ratio is found to be generally high for longer pulses (> 500 µs) generated with MPP due to the fact that the plasma is in a semi-steady state and has had time to evolve. For shorter pulses or non-steady state pulses, peak power transients are apparent. Ionization level and plasma properties can be evaluated on these terms. It was found that near 1 A/cm^2, the plasma characteristics and deposition rate become very similar regardless of the method used to create the pulse.

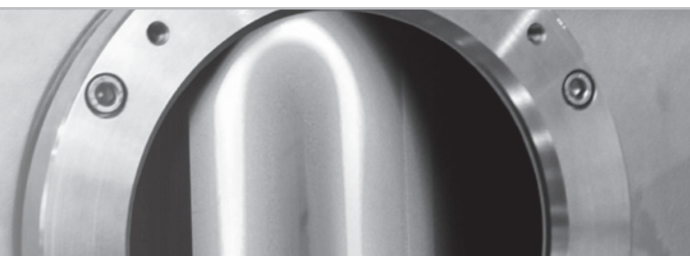


Highly Ionized Pulse Plasma Processes – HIPP Processes

R. Bandorf, G. Bräuer

Fraunhofer-Institute
for Surface Engineering and Thin Films IST
Bienroder Weg 54E
38108 Braunschweig, Germany

Highly ionized pulse plasma processes (HIPP processes), like high power impulse magnetron sputtering (HIPIMS) also known as high power pulse magnetron sputtering (HPPMS), modulated pulse power sputtering (MPP), as well as further modifications involving increased ionization in sputtering have been investigated and matured within the last decade. On European level a concerted action focusing the efforts and bringing together the leading experts was set up and is actually promoting the transition of HIPP processes to industry. This paper will summarize on the different technological developments within more than ten years HIPIMS and familiar technologies. An emphasis will be put on the plasma generation presenting the state of the art in HIPP power supplies ranging from unipolar, bipolar, superimposed HIPIMS to constant current pulsing. Applications using conventional HIPIMS, MPP sputtering, as well as highly ionized gas flow sputtering (HIPP-GFS) and constant current pulsing will be presented. Furthermore examples from academic results up to processes at the stage of industrialization will be presented.



A novel sputtering technique: Inductively Coupled Impulse Sputtering (ICIS)

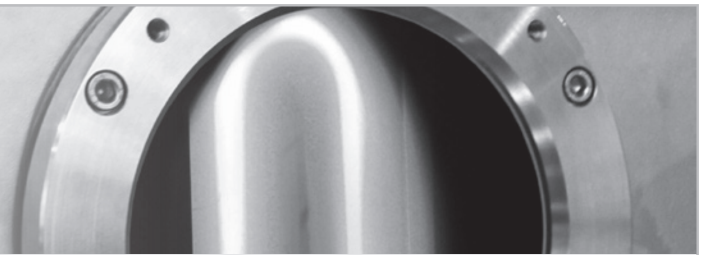
D. Loch*, A. P. Ehiasarian

HIPIMS Technology Centre, Sheffield Hallam University, Sheffield, UK

***Corresponding author**
Email: daniel.a.loch@student.shu.ac.uk
phone: +44 (0) 114 225 4081 | fax: +44 (0) 114 225 3501

Sputtering magnetic materials with magnetron based systems has the disadvantage of field quenching and variation of alloy composition with target erosion. The advantage of eliminating magnetic fields in the chamber is that this enables sputtered particles to move along the electric field more uniformly. Inductively coupled impulse sputtering (ICIS) is a form of high power impulse magnetron sputtering (HIPIMS) without a magnetic field where a high density plasma is produced by a high power radio frequency (RF) coil in order to sputter the target and ionise the metal vapour. In this emerging technology, the effects of power and pressure on the ionisation and deposition process are not known.
The setup comprises of a 13.56 MHz pulsed RF coil operating at a frequency of 500 Hz and a pulse width of 500 µs, which results in a duty cycle of 25 % . A DC voltage of 1900 V was applied to the cathode to attract Argon ions and initiate sputtering. Optical emission spectra (OES) for copper neutrals at a constant pressure of 1.2×10^{-1} mbar show a linear intensity increase for peak RF powers of 500 W – 3500 W and a steep drop of intensity for a power of 4500 W. Argon neutrals show a linear increase for powers of 500 W – 2300 W and a saturation of intensity between 2300 W – 4500 W. The influence of pressure on the process was studied at a constant peak RF power of 2300 W. The intensity of copper and argon neutrals rose linearly for pressures of 2.95×10^{-2} – 1.2×10^{-1} mbar and saturates for pressures from 1.2 – 2.14×10^{-1} mbar. The deposition rate is 99-119 nmh^{-1} for RF-power of 2300 W, average target power of 67 W and a pressure of 1.2×10^{-1} mbar. The microstructure of the coatings shows globular growth at 2.95×10^{-2} mbar and large-grain columnar growth at 1.2×10^{-1} mbar. Bottom coverage of unbiased vias with width 0.360 µm and aspect ratio of 2.5:1 increased from 15 % to 20 % as pressure increased from 2.95×10^{-2} to 1.2×10^{-1} mbar.
The current work has shown that the concept of combining a RF powered coil with a magnet-free HIPIMS powered cathode is feasible and produces very stable plasma. The experiments have shown a significant influence of power and pressure on the plasma and coating microstructure.

Figure 1: Image of an ICIS plasma with a 76.2 mm diameter copper target and 80 mm copper RF-coil.



1 inch-sized HIPIMS glow discharge source

H. Ogiso*, S. Nakano, K. Yukimura

National Institute of Advanced Industrial Science and Technology (AIST), 1-2-1 Namiki, Tsukuba, Japan

***Corresponding author**
E-mail: ogiso.h@aist.go.jp
phone: +81 298 (861) 7267 | fax: +81 298 (861) 2436

A semiconductor fabrication plants becomes significantly bigger in order to realize efficient massproduction of semiconductor devices. At present, the state of art in silicon-wafer size is actually 300 mm, and 450 mm-sized-wafer system is now developing. This flow of the development would result in a high investment-cost. A smaller-sized manufacturing system is more suitable in order to realize a small amount of production in semiconductor devices such as LSI and MEMS chips. A new semiconductor-device manufacturing system has been proposed by AIST and by collaborating companies in Japan, where the devices are fabricated on a 0.5 inch-sized wafer for one chip. This manufacturing system is called »minimal fab«[1]. The »minimal fab« system is promising to reduce the manufacturing cost for a small amount of device fabrication, and the research and development cost as well. The system could also achieve on-demand production of the devices. In order to reduce the size of the manufacturing system, plasma sources have to be also small-sized. On the basis of the background to realize the minimal-fab system, we have been developing a 1 inch-sized magnetron-sputtering plasmasource with keeping a same level of the electrical characteristics such as power density at the target as conventional ones. For efficient operation, we have employed HIPIMS style with a pulsed ionization of sputtered metallic species. **Figure 1** shows a photograph of the unit of the magnetron source with a target diameter of 1-inch (25.4 mm), where the operating conditions for plasma generation are as follows:

Target material: .... titanium
Ambient gas:....................... 4 Pa-argon
Initial target voltage: .... 1 kV (negative polarity)
Duration: .... 170 µs.
Repetition rate:.................. 60 Hz
Current limiting resistance: 5 Ω
Unit container: .... heat-resistant glass
.... (Ø 50 mm × 140 mm)

Figure 2 shows an example of waveforms of the target voltage and the current through the target. The voltage damps to approximately 400 V from the applied initial voltage of 1000 V by initiating a glow plasma with a current of approximately 110 A. The plasma initiates at approximately 12 µs. The plasma area is estimated to be approximately 4 cm^2. Thus, the power consumed at the target is approximately 42 kW, which corresponds to a power density of approximately 10 kW/cm^2.

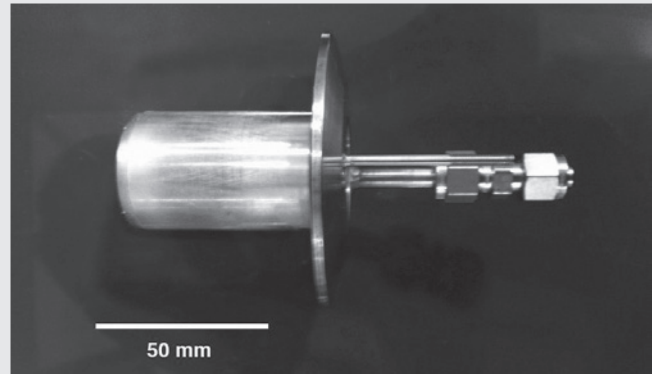


Figure 1: A 1-inch-sized magnetron sputtering plasma source

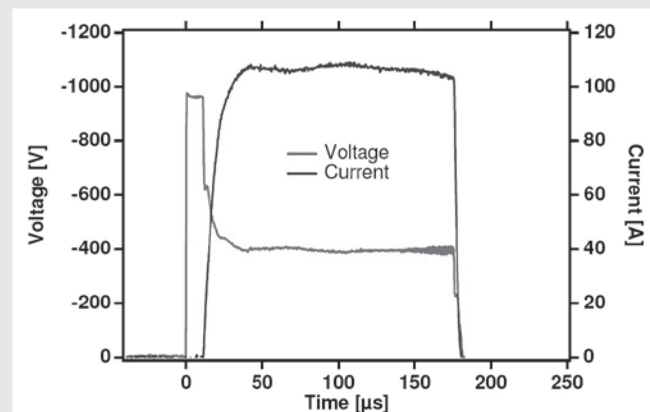
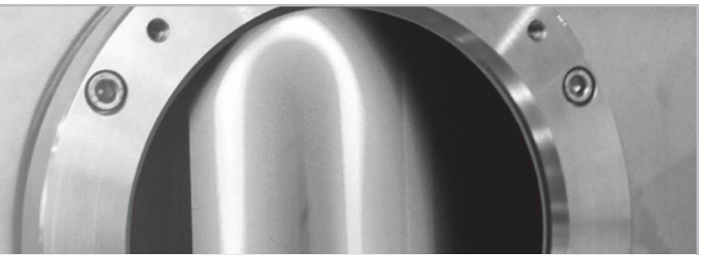


Figure 2: Waveforms of target voltage and current through the target for an initial target voltage of 1 kV with negative polarity and an ambient argon gas pressure of 4 Pa

References:

01. http://unit.aist.go.jp/nano-ele/fabsystem.html

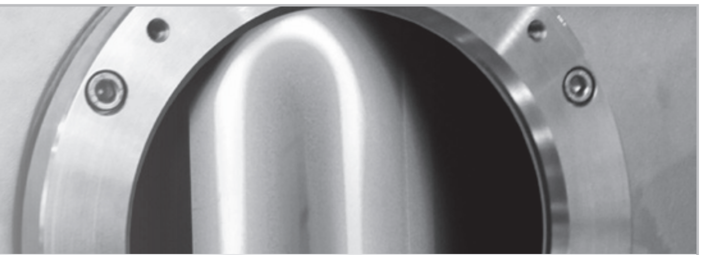


New Method of Generation High Power Pulse Magnetron and Arc Discharges based on oscillatory voltage wave forms

R. Chistyakov, B. Abraham

Zond Inc / Zpulser LLC, Mansfield, Massachusetts, USA

Typically the magnetron discharge current is a function of applied voltage between cathode and anode. In order to generate high power magnetron discharge high voltage should be applied between cathode and anode. A new method of generation of high power magnetron discharge based on application of oscillatory voltage waveforms will be discussed. In this method high power magnetron discharge can be generated by adjusting the frequency of the voltage oscillations and amplitude. The explanation of this phenomenon will be presented. Based on this approach a new HIPIMS plasma generator was developed. The maximum peak power can be in the range of 1.4 MW at maximum output voltage 1700 V. The width of the voltage oscillations is in the range of 3 – 18 µs. The voltage oscillations frequency can be controlled in the range of 10 kHz – 62.5 kHz. The amplitude of the voltage oscillations can be varied by controlling the parameters of the output LC circuit. The voltage current characteristic at different frequencies of voltage oscillations for Cu, C, and Ti discharges will be presented. The application of the oscillatory high voltage waveform for generating near arc free magnetron discharge for reactive sputtering of AlN and Al_2O_3 films will be discussed. The same approach of controlling the frequency of the voltage oscillations can be used for generating high power pulse arc discharge.



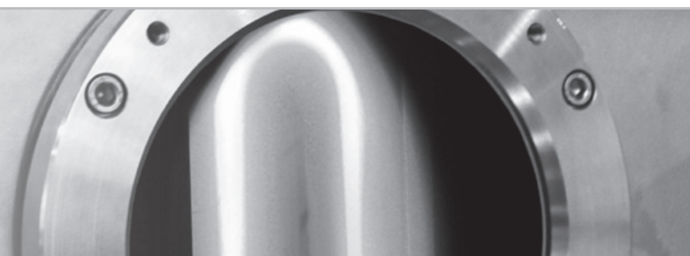
Relation between HIPIMS power supply pulse shape and ion generation efficiency

T. Wallendorf[1], R. Bandorf[2], H. Gerdes[2]

[1] IfU Diagnostic Systems GmbH, Lichtenau, Germany,
twa@ifu.de
phone: +49 37208 889 16 | fax: +49 37208 889 29

[2] Fraunhofer Institute for Surface Engineering and Thin Films IST

A number of HIPIMS processes has been transferred to industry recently or is still under transfer at present. The most interesting aspect of any HIPIMS process is the contribution of charged particles to the film growth. The ion fraction arriving at the substrate is influenced by a number of criteria, e.g. geometry, substrate potential, pulse shape and as supply conditions among others.
In the present paper only the pulse shape influence will be investigated. Several different pulse shapes of a HIPIMS discharge are studied in terms of capability of ion generation efficiency in relation to the power input. A HIPIMS power supply, generating different pulse shapes in a magnetron planar metal target source was investigated using optical emission spectroscopy of the target surface to monitor the different charged particle optical emission intensities during the pulse. The fraction of charged emissions of different pulse shapes are compared. Power input was measured at DC source output and at discharge input. A relation between ion generation capability and energy consumption is derived.



Simultaneous growth rate and film performance based on optimization of the HIPIMS process: A step towards the HIPIMS industrialization

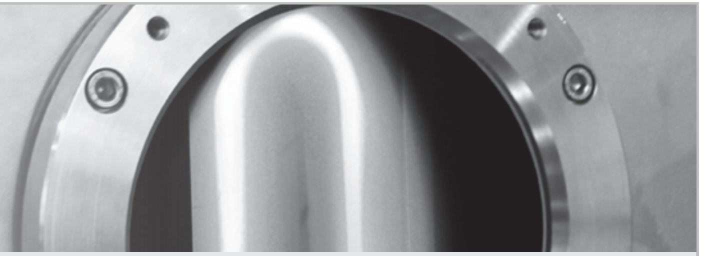
K. Sarakinos*, M. Samuelsson, M. Aiempanakit, U. Helmersson

Plasma & Coatings Physics Division, IFM-Materials Physics, Linköping University,
SE-58183, Linköping, Sweden

***Corresponding author**
E-mail: Kostas@ifm.liu.se
phone: +46 13 281241 | fax: +46 13-287568

High power impulse magnetron sputtering (HIPIMS) has proven to be an elegant way to increase the plasma density in magnetron discharges, thus converting a conventional magnetron source into an ion source. This, in turn, has been shown to enable to tailor the microstructure, the phase, and the atomic composition of the growing films and significantly enhance their density, mechanical and optical performance. At the same time, the high degree of ionization of the sputtered species in combination with the time dependent character of the HIPIMS process have been postulated to be the reason behind the lower film growth rates frequently observed in HIPIMS as compared to conventional magnetron sputtering techniques. This has so far been one of the main obstacles towards the industrialization of HIPIMS. To alleviate this obstacle, significant research effort has been devoted to understand the mechanisms that determine the growth rate in HIPIMS, and to increase its magnitude. During this process, an important fact has often been overlooked: HIPIMS does not represent a single, well defined plasma, but rather corresponds to discharges ranging widely from low ionization dc-like ones, to fully ionized plasmas. Furthermore, one can navigate across the HIPIMS character space, and through proper choice of process parameters, such as pulse repetition frequency, pulse width and power, and magnetic field configuration influence film properties.

In the present talk we contribute towards the industrialization of the HIPIMS process by demonstrating how the above described notion allows for a simultaneous optimization of growth rate and film performance when using HIPIMS. A number of metallic (Cr), metal nitride (CrN) and metal oxide (Al_2O_3, TiO_2) material systems for various applications are used as case studies.

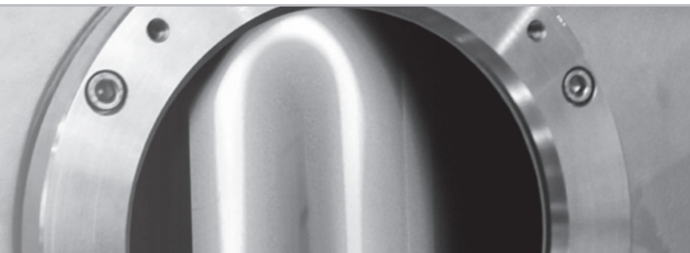


High-rate reactive deposition of non-conductive, highly optically transparent oxide films using high power impulse magnetron sputtering

J. Vlcek, J. Rezek, J. Lazar, T. Koranda

Department of Physics, University of West Bohemia,
Univerzitni 22, 306 14 Plzen, Czech Republic
Phone: +420-377632200 | Fax: +420-377632202
E-mail: vlcek@kfy.zcu.cz

High power impulse magnetron sputtering of zirconium target in argon-oxygen gas mixtures was investigated at a high average target power density in a period, being up to 100 Wcm^{-2}. The repetition frequency was 500 Hz at duty cycles ranging from 2.5 to 10 %. The total pressure of the argon-oxygen gas mixture was around 2 Pa. An effective reactive gas flow control, developed by us, was used for high-rate reactive deposition of non-conductive, highly optically transparent ZrO_2 films. In addition to the ZrO_2 films, high power impulse magnetron sputtering was also successfully used for high-rate reactive deposition of non-conductive, highly optically transparent Al_2O_3 and Ta_2O_5 films. Details of the process and measured properties of the films will be presented.

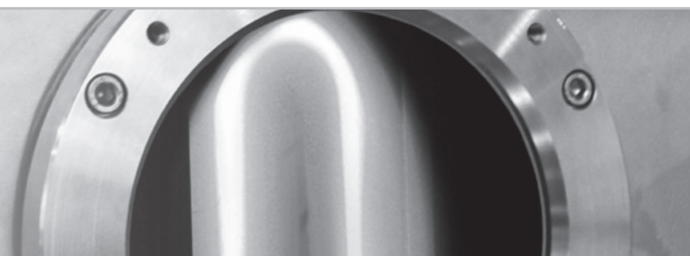


Recent developments on HIPIMS for the deposition of optical functional coatings

M. Vergöhl, S. Bruns, O. Werner, R. Bandorf, G. Bräuer

Fraunhofer Institute for Surface Engineering and Thin Films IST, Bienroder Weg 54E, 38108 Braunschweig, Germany

We report on the latest developments of optical materials such as TiO_2, Al_2O_3, ZnO, Si_3N_4 and ZrO_2 by the use of reactive HIPIMS deposition. Former work has shown that oxygen can have a positive as well as a negative influence on the film properties. By oxygen partial pressure control film properties can be influenced drastically.
Also, it has been shown that with the use of mid-frequency superimposed technique, isolating materials can also be deposited reactively. However due to the broader erosion track of the HiPIMS process planar cathodes suffers still from arcing. Therefore cylindrical magnetron was implemented and HiPIMS deposition was performed in the unipolar mode. We report on first results on the reactive HiPIMS deposition of Alumina coatings.



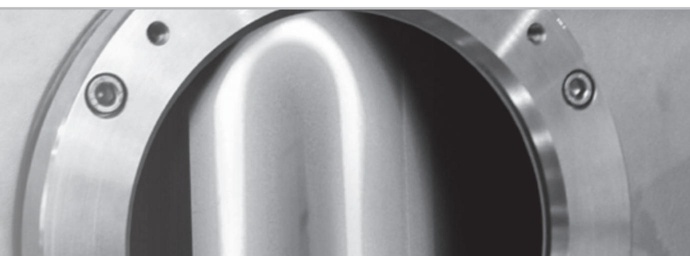
Deposition of TiO_2 crystalline thin films by combination HIPIMS/MF magnetron sputtering system

Z. Hubička*, M. Čada, V. Straňák, Š. Kment, J. Olejníček, L. Jastrabík

Institute of Physics ASCR,
v.v.i. Na Slovance 2 Prague 8 182 21 Czech Republic

***Corresponding author**
hubicka@fzu.cz

Pulsed planar magnetron working in the low frequency HIPIMS together with MF (350 kHz) frequency plasma excitation was used as a plasma source for the deposition of TiO_2 thin films with crystalline phase on unheated substrates. The goal was to characterize the deposition of crystalline TiO_2 films on thermally sensitive substrates. For these experiments, quartz glass SiO_2, glass coated by ITO and polymer foil were used as the substrates. The diameter of titanium target used in the experiments was 50 mm. Ar, O_2 and/or N_2 were used as working gas mixture. The low frequency pulsed source used repetition frequency in the range 100 – 1000 Hz and the active part of the modulation cycle has 100 µs. Maximum current in the pulse was 50 A. Simultaneous MF frequency plasma excitation worked with the frequency 350 kHz with maximum average current 500 mA. Under mentioned conditions, TiO_2 films amorphous or with rutile, anatas or mixture of both structures were deposited on these types of substrates. The formation of particular phases was dependent mainly on the gas pressure, working gas mixture composition and on the ratio of applied power from MF and low frequency power sources. Crystalline structure was observed by Raman scattering and XRD. Chemical composition was measured by XPS. The DC photoconductivity was measured by Van der Pauw methods under illumination of the films by UV radiation. This measurement was done on the films deposited on SiO_2. Photochemical activity was measured on the films deposited on the glass coated by ITO by means of electrochemical cell. Incident photocurrent efficiency was measured in this way for the deposited films. Ion flux and ion energy distribution function at the substrate was measured by the retarding field analyzer SEMION Impedance Ltd. In addition, measurement by time resolved Langmuir probe at the position of the substrate was used as well. Electron energy distribution function and plasma potential time evolution were measured by this technique. These measured plasma parameters differs in the »ON« time of the low frequency pulse when absorbed power from the low frequency HIPIMS source was much higher than delivered power from the MF source and »OFF« time when only MF source was delivering power into the plasma. Emission spectroscopy was also used in order to measure plasma composition for different conditions and investigate relative amount of sputtered Ti and Ti+ and atoms for particular conditions.



Reactive High Power Impulse Magnetron Sputtering of Ti in Ar/O_2 atmosphere

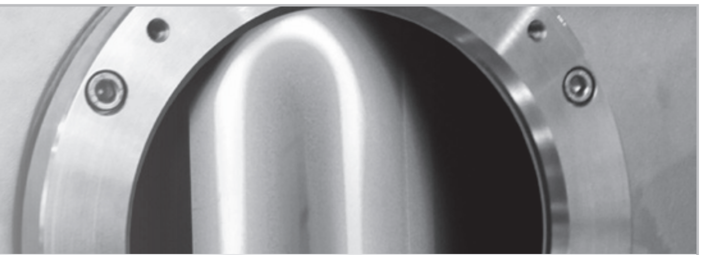
M. Audronis[1], G. Abrasonis[2], R.Heller[2], P. Chapon[3] and V. Bellido-Gonzalez[1]

[1] Gencoa Ltd, Physics Road, Speke, L24 9HP, UK

[2] Division of Nanostructures (FWIO),
Institute of Ion Beam Physics and Materials Research,
Helmholtz-Zentrum Dresden-Rossendorf e.V.,
P.O. Box 51 01 19, 01314 Dresden, Germany

[3] HORIBA Jobin Yvon Sas, 16-rue du canal,
91165 Longjumeau Cedex, France

High Power Impulse Magnetron Sputtering (HIPIMS) is a technologically important Physical Vapour Deposition (PVD) process that is able to provide a highly ionised flux of sputtered species. It is thought to be particularly important for applications where there is a need to coat 3D features. HIPIMS may have other added benefits, as compared to DC or medium frequency (MF) magnetron sputtering, that are related to improved coating structure-property relationship control through self-species ion/plasma assistance. Enhanced structure and properties of PVD thin film materials produced by reactive sputtering are also highly desirable. Significant progress related to providing control means for reactive HIPIMS processes and ensuring stability has been made recently. This paper reports some of the recent process (reactive HIPIMS of Ti in Ar/O_2 atmosphere) and products analysis results. The recently developed Plasma Monitoring based method was used to investigate, monitor and control the deposition process. RBS, ERDA, GDOES and Spectroscopic Ellipsometry were used to characterise the samples produced.



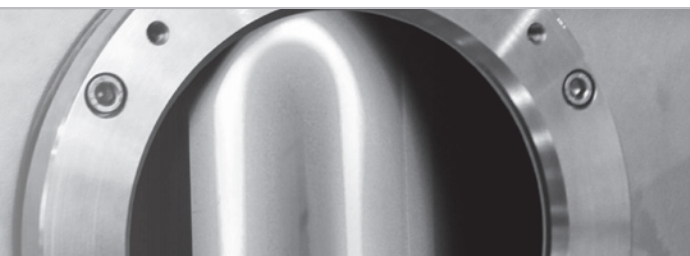
Reactive Sputter Deposition of Alumina Coatings

H. Gerdes, R. Bandorf, D. Loch, G. Bräuer

Fraunhofer-Institute
for Surface Engineering and Thin Films IST
Bienroder Weg 54e
38108 Braunschweig, Germany

Highly ionized pulse plasma processes (HIPP processes) like High Power Impulse Magnetron Sputtering HIPIMS and Modulated Pulse Power MPP have matured in recent years. Actually research focuses increasingly on development for industrial processes. HIPP processes offer a tool for tailoring the film properties and to improve hardness, density, refractive index, and many more properties beyond state of the art. Alumina coatings are used besides application in cutting tools as insulator for electric and sensor applications.

This paper focuses on the process development of an industrial process for deposition of alumina with improved properties regarding the use as insulator. Concerning productivity a high rate deposition process is required for economic production. Therefore the deposition rate must be increased or the film properties improved in a way that thinner films exceed reference films prepared with state of the art technology. Both MPP and HIPIMS, including MF-superposition were investigated and the resulting rates are reported. The films were prepared both without feedback control close to the threshold of the transition to oxide mode and with feedback control. Regarding the insulating properties the films were characterized by their breakdown voltage. MPP films with less than 1 µm thickness showed breakdown voltages up to 1.6 kV. The SEM cross sections of the prepared films showed a dense glassy structure for all the HIPP films.



Structure evolution and wear mechanism in TiAlCN/VCN nanoscale multilayer coatings deposited by reactive High Power Impulse Magnetron Sputtering technology

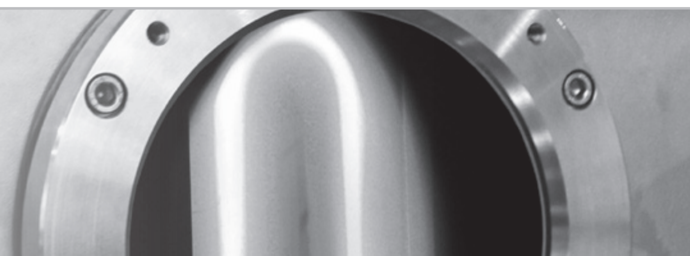
P. E. Hovsepian[1]*, G. Kamath[1], A. P. Ehiasarian[1], I. Petrov[2]

[1] Sheffield Hallam University, Howard Street
Sheffield, S1 1WB, UK

[2] University of Illinois at Urbana-Champaign
104 South Goodwin Avenue, Urbana, Illinois 61801

***corresponding author**
Prof. P. Eh. Hovsepian | e-mail: p.hovsepian@shu.ac.uk

2.5 µm thick TiAlCN/VCN coatings were deposited by reactive HIPIMS process. XTEM showed gradual evolution of the structure of the coating with thickness. The initial structure is nanoscale multilayer with sharp interlayer interfaces. This transforms to nanocomposite of TiAlCN and VCN nanocrystalline grains surrounded by C-rich tissue phase and finally changes to an amorphous carbon rich Me-C phase. In contrast deposition in similar conditions using standard magnetron sputtering produces a well defined nanoscale multilayer structure. Depth profiling by AES showed that the carbon content in the HIPIMS coating gradually increased from 27 % at the coating substrate interface to 35 % at the top thus supporting the TEM observations.
Energy-resolved mass spectrometry revealed that HIPIMS plasma is a factor of 10 richer in C^{1+} ions, and therefore more reactive, as compared to the plasma generated by standard magnetron discharge at the same conditions. The peculiar structure evolution in HIPIMS is discussed in relation to target poisoning effect and carbon outward diffusion during coating growth.
Highly abrasive AlSi9Cu1 alloy was dry machined using TiAlCN/VCN coated 25 mm diameter end mills to investigate the coating-work piece material interaction.
Green (532 nm excitation) and UV (325 nm excitation) Raman spectroscopy was employed to identify the phase composition of the built up material on the cutting edge and swarf surfaces produced during machining. These analyses revealed formation of lubricious Magneli phases namely V_2O_5 and graphitic carbon as well as highly abrasive SiO_2 and Al_2O_3 thus shedding light on the wear processes and coating tribological behaviour during machining.



Deposition of chromium and chromium nitride using DC and fast-HIPIMS discharges

A. Ferrec*[1], M. Ganciu[2], M. A. Djouadi[1], P. Y. Jouan[1]

[1] Université de Nantes, UMR CNRS 6502, Institut des Matériaux Jean Rouxel, 2 rue de la Houssinière – 44322 Nantes, France

[2] National Institute for Laser, Plasma and Radiation Physics, Plasma Department, 077125, Magurele, Bucharest, Romania

***Corresponding author**
E-mail: axel.ferrec@cnrs-imn.fr
phone: 00 33 (0)2 40 37 39 67

Principally for its corrosion resistance and its mechanical behavior, chromium nitride was widely studied and developed by classical magnetron sputtering[1] and more recently to improve it, by HIPIMS over the last decade[2,3]. Here, a comparison of the microstructure and the mechanical properties was investigated using conventional DC and fast-HIPIMS discharges[4]. This is a new technology based on a pre-ionisation system and allows us to work with pulses of few microseconds, while remaining in very stable conditions and avoiding the arc formation. In this study, the range of the pulse width was varied between 10 and 40 µs and we have operated with a frequency between 80 and 1000 Hz. Although the dc and HIPIMS parameters are not really comparable, we produced the films with the same average power. We characterized the layers with X-ray diffraction and the HIPIMS films revealed a best cristallinity in both Bragg Brentano and rocking curve mode. The results extracted from the SEM pictures revealed a higher density and a smaller roughness for the HIPIMS films. With this kind of process it is already well known that there is a drop of the deposition rate. But to compare more precisely the two methods, we chose to focus on the film density, determined by reflectometry, which is one of the most important parameter for mechanical applications. In the reactive mode, we have also measured the effect of the nitrogen flux on the HIPIMS discharge electrical characteristics. The mechanical properties such as hardness, have been characterised by nanoindentation tests.
Keywords Fast HIPIMS, DC sputtering, Chromium nitride

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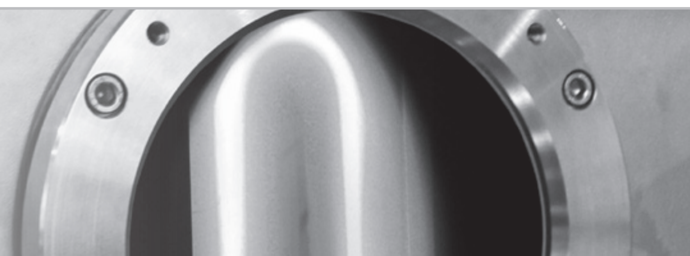

Syntheses and characterisation of TiC and TiC/a-C nanocomposite using DC magnetron sputtering and high power pulse magnetron sputtering

L. Zottarel[1,*], M. Colasuonno[1], A. Surpi[1], M. Bazzan[2], N. Argiolas[2], A. Patelli[1]

[1] Associazione CIVEN, via delle Industrie 5, I-30175 Venezia, Italy

[2] Università di Padova, Dipartimento di Fisica, Padova, Italy

***Corresponding author**
E-Mail: Zottarel@civen.org
phone: +39 041 509 3929 | fax: +39 041 509 3988

Nanocomposite thin films have recently attracted much interest because of the possibility of synthesis of materials with unique properties, e.g. super-hardness, combined hardness and low frictions[1]. Nanocomposite TiC/a-C coatings based on of TiC nanocrystallites dispersed in solid lubricant matrix like amorphous carbon (a-C) or amorphous hydrocarbon (a-C:H) have been shown to enhance the hardness and toughness while maintaining the low coefficient of friction[2-3]. Furthermore, high power pulse magnetron sputtering (HIPIMS) has been recently proposed to enhance toughness and adhesion of these materials to metal substrates. The aim of this study is to thoroughly investigate the effect of different power supplies on the composition and the mechanical properties of TiC/a-C nanocomposites deposited by PVD magnetron sputtering. Both HIPIMS and traditional DC magnetron sputtering (DCMS) were used as power supply for a TiC target in pure Argon atmosphere. Further graphite targets were used to tailor the final composition of coatings. Using this configuration a set of samples were deposited both on silicon, high strength aluminium alloy and high speed steel substrates to verify the mechanical properties and coating adhesion as a function of substrate material.
The effect of using HIPIMS and traditional DC power supply were investigated and discussed. All the coatings, have been structurally characterised by XRD, morphologically by cross-sectional SEM, compositionally by α-2MeV RBS analysis. Hardness and Young modulus have been measured by nanoindentation; coating adhesion by microscratch test Structural and compositional characterizations were carried out by XRD and α-2MeV RBS techniques respectively. The coating morphology have been evaluated by cross sectional SEM. Nanoindentation was performed to evaluate hardness and elastic modulus while microscratch test was used to evaluate the coating adhesion.

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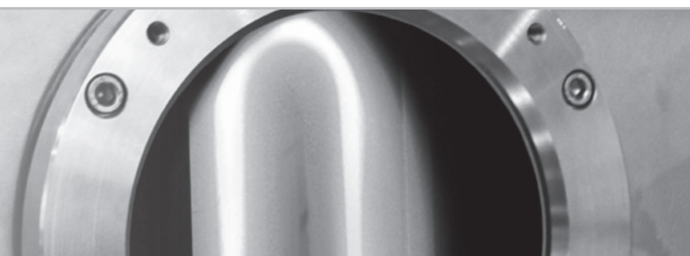


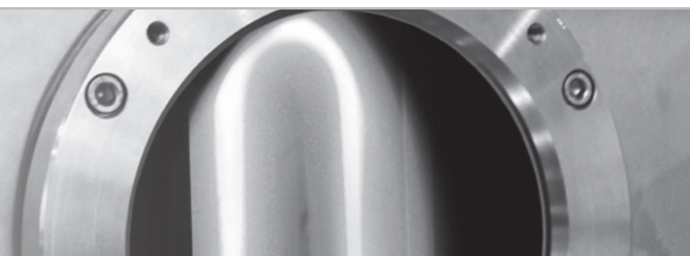
Towards synthesizing high density and sp^3 rich carbon films using high power impulse magnetron sputtering

A. Aijaz*, K. Sarakinos, D. Lundin, U. Helmersson

Plasma and Coatings Physics Division, IFM-Materials Physics, Linköping University, SE-581 83 Linköping, Sweden

***corresponding author**
asim@ifm.liu.se

Hydrogen free amorphous carbon (a-C) films exhibit unique and adjustable mechanical, electrical, and optical properties ranging from those of graphite to those of diamond finding its applications ranging from lubrications to ultra dense hard coatings. These properties are controlled by the hybridization state of carbon atoms i.e. the ratio of the sp^3 to sp^2 hybridized bonds. The hybridization state in a-C films grown by physical vapour deposition (PVD) techniques is controlled by energy and flux of ionized carbon as well as buffer gas species through the subplantation and densification processes. The ion energy can be manipulated, for example, by applying a bias potential to the substrate. The state of the art approaches for high density and sp^3 rich carbon films include ionized PVD techniques such as filtered cathodic vacuum arc and pulsed laser deposition. These techniques provide dense films with up to 90 % of sp^3 fraction (also referred to as tetrahedral amorphous carbon). They suffer, however, from low film growth rates and lack of lateral uniformity and upscalability potential. This issue can be solved using magnetron sputtering. However, magnetron sputtering does not yield high enough ionization of the carbon to allow for growth of films with a high sp^3 content.
High power impulse magnetron sputtering (HIPIMS) has shown an enhancement of the degree of ionization for most of metals and hence improved properties of the films. However, carbon having a higher ionization potential (higher as compared to most metals) still poses a great challenge. In the present study we address the low degree of ionization of carbon in a magnetron discharge. The resulting films, grown at a negative substrate bias ranging from 0 to -150 V show a density, obtained by high resolution x-ray reflectivity measurements, as high as approx. 2.8 g/cm^3. Typical Raman spectroscopic parameters such as D-peak to G-peak ratio (I(D)/I(G)) and full width at half maximum of G-peak (FWHM (G)) show a prominent difference in the films grown with our new process as compared to the conventional HIPIMS carbon discharge.



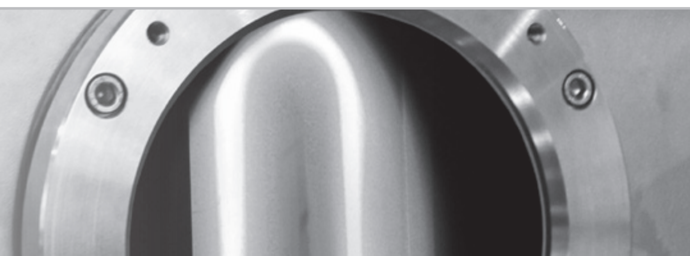
High Power Impulse Magnetron Sputtering Discharges: Instabilities and Plasma Self-Organisation

A. Hecimovic[2], A. P. Ehiasarian[1], J. Winter[2], T. de los Arcos[2], R. New[1], V. Schulz-von der Gathen[2], M. Böke[2]

[1] HIPIMS Technology Centre, Sheffield-Hallam University, UK

[2] Institute of Experimental Physics II, Ruhr-University Bochum, Germany

We report for the first time on instabilities of High Power Impulse Magnetron Plasmas (HIPIMS) which are likely to be of the generalised drift wave type. They are characterized by well defined regions of high and low plasma emissivity along the racetrack of the magnetron. The azimuthal mode number m and speed depend on plasma current and density, on the pressure and atomic mass of the working gas. The structures rotate in E × B direction at frequencies up to 200 kHz. Collisions with residual gas atoms slow the rotating wave down and reduce the instability wavelength, whereas the ionisation degree of the gas accelerates it and increases the instability wavelength. The instabilities result not only in intermittent modulated particle fluxes away from the cathode but most likely also in highly localized transient heat loads on the cathode surface, which may be of significant technological importance.



Time-resolved investigation of hybrid dual-HIPIMS discharge during deposition of intermetallic Ti-Cu films

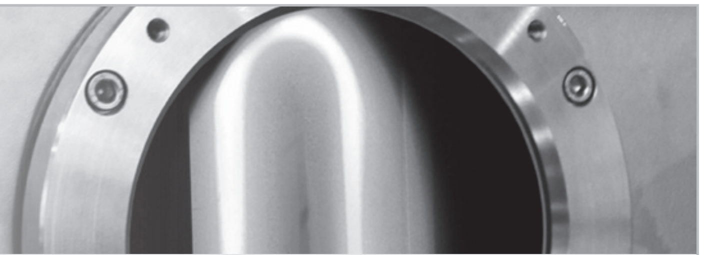
V. Straňák[2]*, Z. Hubička[2], S. Drache[1], M. Čada[2], R. Bogdanowicz[1], H. Wulff[1], R. Hippler[1]

[1] University of Greifswald, Institute of Physics, Felix-Hausdorff-Str. 6, 17489 Greifswald, Germany

[2] Academy of Sciences of the Czech Rep., Institute of Physics, Na Slovance 2, 18221 Prague 8, Czech Republic

***Corresponding Author**
Vitezslav Stranak
Institute of Physics AS CR, Na Slovance 2, 18221 Prague 8, Czech Republic
E-Mail: stranak@fzu.cz
phone: +420 266 052 127, fax: +420 286 581 448

DC-pulse magnetron sputtering has become an important deposition technique in last two decades. In this way, higher plasma density in pulse can be achieved. Furthermore, other important parameters like electron and ion energy distribution functions, ion and thermal fluxes on the substrate can be strongly affected and used for controlling of deposition process. The modulated in mid-frequency (5-350 kHz) cathode voltage with duty cycle of typically 50 % usually results in high thermal energy flux and low (or comparable with DC-sputtering) ionization degree, ion energy and ion flux. On the contrary, the ion flux towards the substrate is typically high in HIPIMS (modulated with low repetition frequencies 50-500 Hz and short duty cycle of < 1 %) and can increase density and homogeneity of deposited thin films. However, because of high energies and powers a large fraction of the sputtered particles is preferentially ionized which reduces the deposition rate during HIPIMS. There have been already published some works focused on increasing of the deposition rate while at the same time the advantages of HIPIMS are preserved: slight increase of repetition frequency, longer pulse widths, employing alternately working magnetrons in so-called »dual« configuration, combination of single bipolar-HIPIMS discharge with bipolar high frequency, etc. In this work, we introduce hybrid-dual-HIPIMS system for deposition of intermetallic Ti-Cu films with antimicrobial effects. Dual-HIPIMS consist of two independent HIPIMS loops alternately employing two sputtering sources as cathode-anode and vice versa. The hybrid system is based on a parallel combination of dual-HIPIMS (repetition frequency f_H = 100 Hz, pulse width T_H = 100 µs) and dual mid-frequency discharge (MF, repetition frequency f_{MF} = 94 kHz, pulse width T_{MF} = 3 µs). The MF discharge, running during the idle time of HIPIMS, supports re-ignition of dual-HIPIMS pulses. The excitation of Ar and metal particles during HIPIMS pulses was studied by time-resolved optical emission spectroscopy and time-resolved optical emission imaging. Time resolved diagnostics of ion flux and measurements of ion velocity distribution function (IVDF) by retarding field analyzer (RFA) combined with measurements of total energy flux (calorimetric probe) were carried out with respect of thin film formation. Thin film properties were investigated, too. Crystallographic phases of deposited films were diagnosed by grazing incidence x-ray diffractometry (XRD), chemical composition was measured by x-ray photoelectron spectroscopy (XPS). The combination of these diagnostic methods enables an extensive characterization of discharge and its effect on film properties.
It was observed that combination of MF and dual-HIPIMS discharges allows: (i) significant decrease of working pressure in the vacuum chamber, (ii) control of specific film properties, e.g. lattice size, (iii) improved re-ignition of dual-HIPIMS pulses, i.e. faster re-ignition and larger HIPIMS peak currents.



Direct energy influx measurements in High Power Impulse Magnetron Sputtering

P. A. Cormier[1], A. Balhamri[2], A. L. Thomann[1], S. Konstantinidis[2], R. Dusart[1], N. Semmar[1], J. Mathias[1]

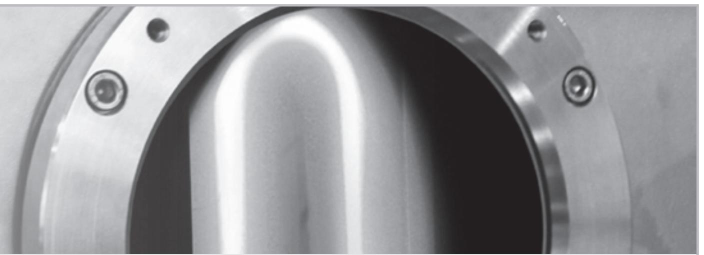
[1] GREMI, Université d'Orléans, 14 rue d'Issoudun, B.P. 6744, 45067 ORLEANS Cedex 2, France
E-Mail: pierre-antoine.cormier@univ-orleans.fr

[2] ChIPS, Université de Mons, Université de Mons, 1 Avenue Nicolas Copernic, 7000 Mons, Belgique

High Power Impulse Magnetron Sputtering (HIPIMS) is an impressive improvement of conventionnal magnetron sputtering. Lots of work have been done around plasma diagnostic and the characterization of deposited thin films. HIPIMS generates high density plasmas and exhibits dense microstructure. Particular properties of HIPIMS deposited thin films are directly influenced by the energetic conditions at the substrate surface.

The knowledge of the energy balance at the substrate surface is necessary for the understanding of thin film growth. Most of studies focused on this point are based on indirect energy influx measurement. These techniques involve measurements uncertainty because of the signal treatment.

We developed a heat flux diagnostic based on a commercial heat flux microsensor (HFM) for the direct measurement of the energy influx, based on the Seebeck effect. The HFM consits of a thermopile with 1 ms time resolution. The diagnostic was placed in front of balanced and unbalanced magnetron cathode to study the influence of magnetic field configuration on the mean energy influx at the substrate surface. As expected, first results suggest that energy influx is higher in the case of unbalanced HIPIMS. The effect of the pulse time duration on the mean energy influx was also investigated. It results, the time duration of interaction between the HIPIMS plasma.



Measuring the plasma potential of HIPIMS discharges

A. Rauch, J. M. Sanders, R. Mendelsberg, A. Anders*

Lawrence Berkeley National Laboratory,
Berkeley, California

***corresponding author**
1 Cyclotron Rd, MS 53, Berkeley, CA 94720, USA
phone: +1-510-486-6745 | Fax +1-510-486-4374
E-Mail: aanders@lbl.gov

The potential distribution in front of a magnetron target is self-consistently regulating the fluxes of charged particles and associated energy of the discharge. It is known that most of the potential difference drops in a thin sheath adjacent to the target, and a much smaller fraction drops in a magnetic presheath whose structure is determined by the shape of the magnetic field. There are only a few direct measurements of the plasma potential of magnetrons, and even fewer for operation under HIPIMS conditions. This lack is not related to a possible absence of interest but due to the technical difficulties of measuring plasma potential.

Researchers often limit measurements to the floating potential, and/or limit the studies to stationary plasmas. In this contribution we present data obtained by an emissive probe specially adapted to HIPIMS operation, and consider the case of argon / niobium plasma made below and above the threshold of self-sputtering runaway. We consider the case of short (pulse length < 200 µs) and long (> 200 µs) pulses, the former being characterized by a triangular current pulse shape, and the latter reaching steady-state at a relatively high current level (50 – 100 A), depending on the applied voltage and other factors. We explain the probe principle, some of its innovative features compared to conventional emissive probes, and apply the technique to map the potential distribution as a function of discharge conditions. An example of such measurements is shown in Fig. 1, where the emissive probe was moved to different positions parallel to the target surface at a fixed distance (z) from it. We expect that a comprehensive data set will help us to elucidate ion fluxes and operation of the HIPIMS discharge.

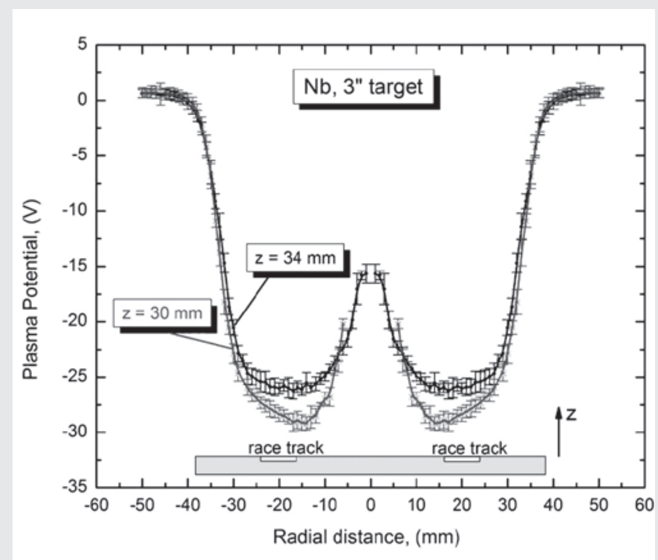
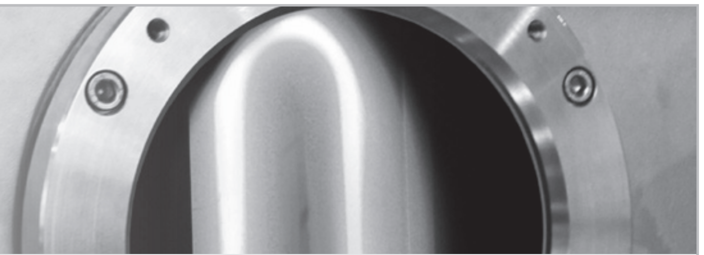


Fig. 1. Example of plasma potential measurements (with respect to the grounded anode) over a 3" niobium target for two distances from the target surface. The measurements were done over one half of the target; the other side was simply mirrored for easier understanding. The data refer to the potential established in the steady-state phase after gas rarefaction. Each point is the average of 10 measurements, and the error bars indicate the standard deviation. The discharge conditions were as follows: Ar pressure 0.3 Pa, applied voltage 380 V for 400 µs using a SPIK2000A pulser, current in steady-state phase 50 A, pulse repetition rate 70 p.p.s., average power 300 W (indicated at charging unit).



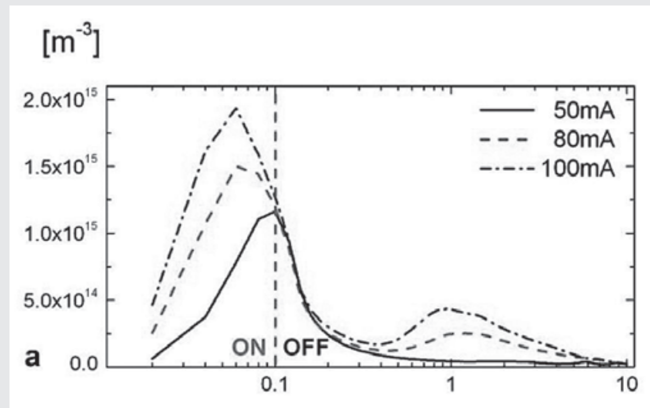
Time-resolved investigation of Ar* density and temperature in HIPIMS discharge by means of tune diode laser absorption spectroscopy

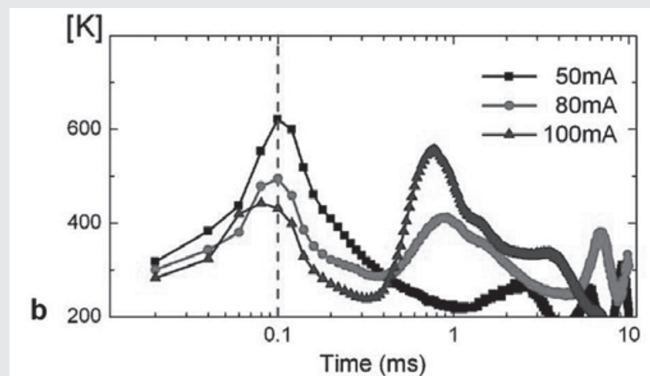
H.T. DO[1], M. ČADA[2,*], V. SUSHKOV[1], Z. HUBIČKA[2], R. HIPPLER[1]

[1] UNIVERSITY OF GREIFSWALD, INSTITUTE OF PHYSICS,
FELIX-HAUSDORFF-STR. 6, 17489 GREIFSWALD, GERMANY

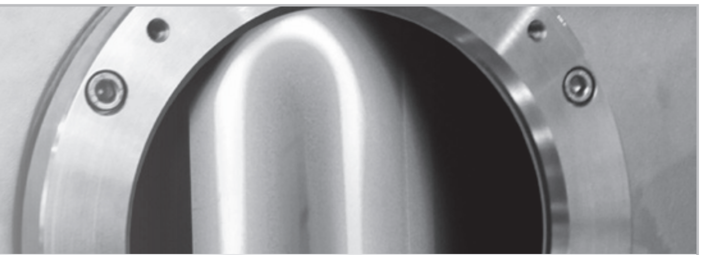
[2] ACADEMY OF SCIENCES OF THE CZECH REP., INSTITUTE OF PHYSICS, NA SLOVANCE 2, 18221 PRAGUE 8, CZECH REPUBLIC

***CORRESPONDING AUTHOR**
MARTIN ČADA
INSTITUTE OF PHYSICS AS CR, NA SLOVANCE 2,
18221 PRAGUE 8, CZECH REPUBLIC
E-MAIL: CADA@FZU.CZ
PHONE: +420-266052995 | FAX: +420-286581448





Time-resolved tune diode laser absorption spectroscopy has been used for Ar metastable density and temperature measurement. The Lesker Torus planar balanced 2“ diameter magnetron equipped with titanium target operated in pure Ar atmosphere at pressure 4 Pa. A magnetron discharge was driven using a pulsed power supply. The repetition frequency of pulsed discharge was held at 2.5 kHz or at 100 Hz. The active plasma pulse time was set on 100 µs for all our experiments. The mean current was set to range ID = 50 – 500 mA. In the HIPIMS mode, two peak structure were observed in both Ar metastable density (panel **a** of figure) and temperature (panel **b** of figure) time evolution. One peak appeared during the pulse on-time and second in the pulse off-time. However, we found a noticeable difference between them. The temperature peak in the pulse on-time appears after the density peak, while the behaviour is inverse in the pulse off-time. We believe that this phenomenon is due to the difference in production mechanism of Ar metastable atoms. In the active plasma pulse, the metastable atoms are excited from ground state of »cold« atoms by electron impact excitation. But in the afterglow, metastable atoms are generated from »hot« ions by recombination processes. The time-resolved Langmuir probe measurements were carried out at position where laser beam passes through the plasma to back up our hypothesis. Calculated plasma parameters were used for modelling of the temporal variation of metastable density.



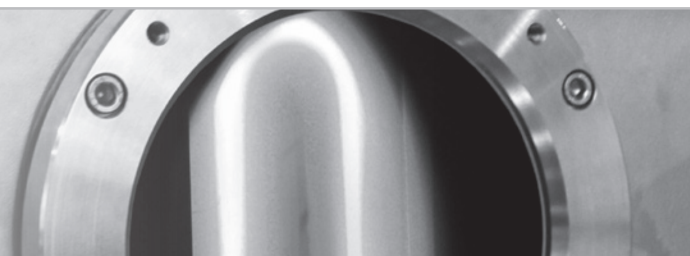
Time Resolved Optical Emission Studies of Pulsed Magnetron Discharges

M. Bilek, A. Weeks-Ross, B. Treverrow and D. R. McKenzie

School of Physics, A28, University of Sydney, NSW, 2006, Australia,

Pulsed plasmas have a number of advantages over their continuous mode counterparts for materials synthesis applications. A major operational advantage is that the pulsed mode is much less affected by problems associated with instabilities, surface charging of substrates and cathode or target poisoning and as such typically present wider process windows. Pulsed plasmas are transient, operating far from equilibrium at some stages in the pulse, and often showing an evolution of parameters throughout the pulse. Resolving the transient phenomena and determining their effects on plasma conditions at the substrate is a prerequisite for achieving good control over film deposition.

In this paper, we employ optical plasma diagnostics to study the evolution of species in a high powered pulsed magnetron discharge. Our multi-source sputtering system is driven by a pulsed power supply capable of producing constant voltage pulses up to 1000 V at currents up to 450 A. Our time resolved optical emission studies show that high charge states evolve throughout the pulse with the mean charge state peaking at the end of the pulse. Differences between the evolution of the first ionised state and that of the higher charge states indicate that distinct electron populations may be responsible for their creation: one characterised by LTE and a temperature close to 1 eV while the other containing fast secondary electrons accelerated through the sheath.
Enhancements of the average charge state and average discharge current achievable by pulse fragmentation will be demonstrated and discussed. A brief comparison with the behaviour of the charge state distribution in a pulsed cathodic arc will also be presented.



Comparison of HIPIMS process technologies for deposition of bendable ITO films

B. Szyszka*, V. Sittinger, W. Werner, M. Vergöhl, J. Mahrholz, R. Bandorf

Fraunhofer Institute for Surface Engineering and Thin Films IST, Bienroder Weg 54E, 38108 Braunschweig, Germany

*Corresponding author
bernd.szyszka@ist.fraunhofer.de

The utilization of HIPIMS processes for oxide film deposition has been subject of intense R&D work in the last couple of years. Issues such as dense and smooth films, increased index of refraction, improved conductivity and durability have been the driving the forces for these developments. Substantial improvements have been achieved in the field of bendable ITO films applicable for ice free window applications[1]. Achieving crystalline growth on unheated substrates turned out to be the crucial point for crack free bending and proper mechanical stability of the films[2]. The underlying HIPIMS process based on Advanced Energy laboratory equipment at that time was tailored in a way to achieve discharge current in the order of 1000 A where short pulse time in the order of 30 µs have been necessary to limit arcing to a tolerable level. Therefore, the steep rise of discharge current is of major concern for this application.
This paper addresses the process transfer of our patented ITO HIPIMS process towards more industrial style equipment. We investigate into the applicability of various HIPIMS power supplies to obtain crystalline ITO films at room temperature and we analyze the processes in terms of arcing behavior and process stability.
Understanding and tailoring the effect of sputtered materials ionization and intense plasma activation by fast ion bombardment will be a key factor for further material development to transfer the promising results for HIPIMS ITO also to other classes of TCOs and dielectric films.

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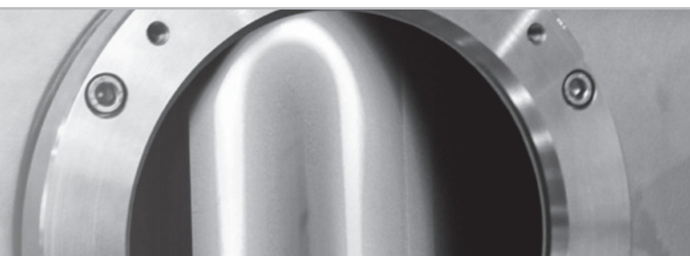

Reactive HIPIMS with auxiliary Al electrode for ZnO:Al thin film deposition

V. Tiron*, C. Costin, L. Sirghi, G. Popa

Alexandru Ioan Cuza University, Faculty of Physics,
Iasi, 700506, Romania

***Corresponding author**
Dr. Vasile Tiron
phone: +40 232 201188 | fax: +40 232 201150
E-Mail: vasile.tiron@uaic.ro

For most optoelectronic devices, such as flat panel displays and solar cells, it is essential to use a transparent electrode, usually consisting of a thin film of transparent conducting oxide. ZnO:Al is a potential candidate for transparent electrodes in optoelectronic device due to the high transmittance in visible region and low resistivity.
By controlling the doping level the electrical properties can be changed from n-type semiconductor to metal while maintaining optical transparency.
A new technique is proposed for precise doping control of ZnO:Al thin films deposited in reactive High Power Impulse Magnetron Sputtering. An auxiliary aluminum electrode was added to a reactive Ar/O_2 pulsed magnetron with planar Zn target (5.6 cm in diameter) in order to obtain a controlled doping of ZnO. The system is characterized by very short discharge pulses (1-10 µs) attaining a maximum cathode current of about 50 A for a maximum applied voltage of 1 kV with a pulse repetition frequency of about 1 kHz[1]. The HIPIMS plasma was characterized on-pulse and post-discharge using time-resolved optical emission and absorption spectroscopy and monitoring the ion current flowing through the auxiliary aluminum electrode. Al neutral density in gas phase has been controlled by the discharge current and the biasing voltage on the auxiliary electrode (which influence the ion bombardment of the electrode) and measured by laser resonant absorption spectroscopy. The fraction of Al dopant in the deposited films was estimated by X-ray Photoelectron Spectroscopy (XPS) measurements. The goal of this work was to correlate Al density measured in the gas phase with Al concentration in the deposited films. It was found that the obtained aluminum concentration in the deposited films was strongly dependent on the auxiliary electrode bias, oxygen/argon gas ratio and relative position of target-electrode-substrate. It was also investigated the effect of the aluminum concentration on the structural, electrical and optical properties of AZO thin films deposited by HIPIMS. The film surface topography has been characterized by atomic force microscopy (AFM), while X-ray Photoelectron Spectroscopy (XPS) and X-ray difractometry (XRD) were used to examine the internal microstructure of the deposited films. The optical properties of the deposited films were studied by UV/VIS and photoluminescence spectroscopy.

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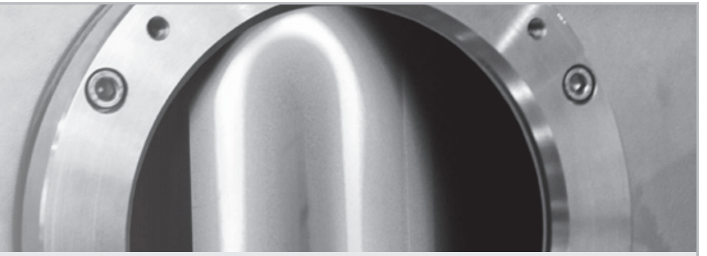


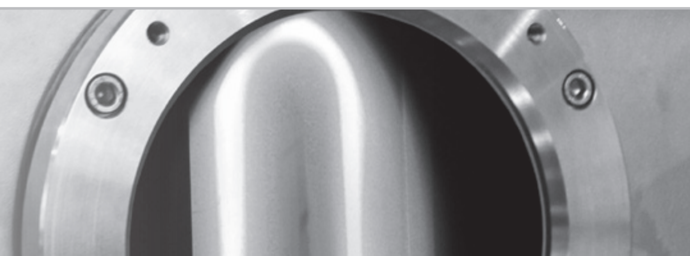
Pulse Magnetron Sputtering with high power density – trial of a critical evaluation

P. Frach*, C. Gottfried, F. Fietzke, H. Klostermann

Fraunhofer-Institute for Electron Beam and Plasma Technology FEP, Dresden

***corresponding author**
phone: +49(0)351-2586-370 | fax: +49(0)351-2586-55-370
peter.frach@fep.fraunhofer.de

In this paper specific advantages and disadvantages of different pulse magnetron sputtering processes (unipolar, bipolar and HIPIMS) as well as current and potential fields of application will be discussed. On the example of TiO_2 the typical effects and their influence on film properties occurring during the transition from classical medium frequency pulse magnetron sputtering to high energy pulse sputtering will be described. The discharge current density was varied between 0.2 and 3.5 A/cm^2. Aspects of energy feed-in and reactive process control in the transition mode will be considered. Furthermore the influence of rising ionisation on the occurrence of crystalline phases and on mechanical, optical and photocatalytic properties of the layers will be presented.
The paper concludes with a placement of the processes related to other PVD-processes that is based on further own experimental results and evaluation of dependencies as well as considering published results of other groups regarding pulse magnetron sputter processes of high power density for the deposition of hard coatings and TCO.

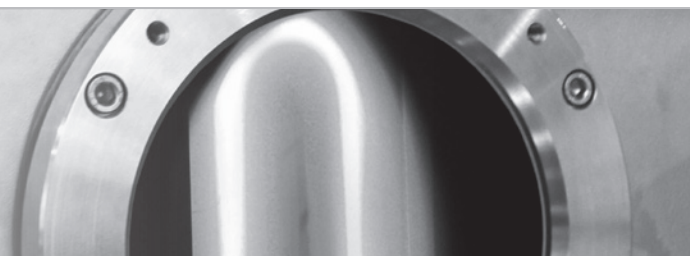


Obtaining coatings with 3-1-2 (Ti-Si-C) stoichiometry from a Ti_3SiC_2 compound target by HIPIMS

M. Balzer, M. Fenker

fem – Research institute for precious metals and metals chemistry, Katharinenstrasse 17, 73525 Schwäbisch Gmünd, Germany
phone: +49 (0)7171 – 1006 31 | fax: +49 (0)7171 – 1006 54

When MAX-phase thin films shall be produced by magnetron sputtering three major problems have to be solved: a) The substrate itself or an interlayer has to exhibit a suitable crystal structure allowing epitaxial MAX-phase growth, b) a high substrate temperature is necessary for phase nucleation and c) the particles have to arrive at the substrate with the appropriate stoichiometry.
In literature it is reported that for depositing the Ti_3SiC_2 MAX-phase a) single crystal oxides like Al_2O_3 (0001) and MgO (111) with TiC (111) interlayers are used as substrates and b) a substrate temperature above 800 °C is necessary. Obtaining the stoichiometry by sputtering from a Ti_3SiC_2 compound target was reported to be challenging due to the demixing of species on their path to the substrate. The coatings were reported to contain too much C and too less Ti, which was tried to overcome e.g. by using an additional Ti-Source. In this work a Ti_3SiC_2 compound target, driven by a HIPIMS power supply, has been used to investigate the possibilities to overcome this problem by providing ionisation and higher kinetic energy to the particles. Flat substrates of 10 cm in length were fixed in front of the 2” target and no additional substrate heating was applied because the film stoichiometry was the only matter to be analysed. The deposition parameters varied were: Target-substrate-distance, Ar-pressure, substrate bias and HIPIMS pulse length. The film stoichiometry was measured at several axial distances from the point straight opposite of the target centre by using GDOS calibrated EDX. In order to gain a reliable base the GDOS again was calibrated beforehand by measuring the Ti_3SiC_2 target with its known composition. It was found, that in the case of Ti_3SiC_2 the element specific ionisation degrees generated by HIPIMS plasma can effectively help solving the demixing problems caused by the different mean free paths of the species. A film stoichiometry very close to the desired 3-1-2 was found for several parameter combinations.



Deposition of transparent nickel oxide by reactive Fast-HIPIMS

A. Ferrec, A. Karpinski, D.T. Nguyen, N. Ouldhamadouche, M. Richard-Plouet, L. Cattin, M. Ganciu, L. Brohan, M. A. Djouadi, P. Y. Jouan*

Institut des Matériaux Jean Rouxel (UMR 6502), 2 rue de la Houssinière, BP 32229, 44322 Nantes, Cedex 3, France

***Corresponding author**
E-mail : pierre-yves.jouan@cnrs-imn.fr
phone: 00 33 (0) 240-376445 | fax : 00 33 (0) 240-373959

The organic photovoltaic cells (OPV) up to now convert 5 % of the solar spectrum and last reports predict that efficiencies as high as 10 % can be achieved. For the classical OPV, charge carrier transport from the photosensitive polymer is performed by incorporation of organic semiconductors which are in contact with metallic electrodes. The interfaces between metal and organic components as well as the natures of organic semiconductors play an important role for the effective charge transport and degradation processes. Limitating the sensitivity of polymer materials becomes the path for development of hybrid OPV. One possible way is to use inorganic semiconductors. Metal oxide semiconductors as protective layers between metal electrodes and polymers, are interesting candidates for achieving charge transport and avoiding the organic-metal interfaces. We propose the NiO as p-type semi-conductor to ensure charge carriers and electron/hole blocking layers. The characteristic wide gap energy permit one to achieve high transparency for thin films of NiO. We have deposited transparent p-type semiconductive NiO thin films on the conductive glass by reactive fast-HIPIMS. The characterization of the films, show that depending on the oxygen content, NiO grows along preferential orientations: either the most dense [111] direction for low oxygen percent or [200] for higher oxygen content. We have highlighted two regimes of fast-HIPIMS[1] discharge. The first one could be called classical one with a peak current around 15 A during the pulse and a deposition rate close to the DC mode. The second one shows a very high current peak, up to 60 A, during only 4 µs and a very low deposition rate. In this case, the target is poisoned without sputtering because all energy is devoted to the ejection of secondary electrons and ionization of Ni, O_2 and O atoms, which will return to the target. This produces an abrupt increase of the current that could be motivated by the passage to an ionised and neutral oxygen cycle between magnetised plasma and cathode explaining thus the very low deposition rate. It has been measured that O_2+ and O+ species are preferentially present at the cathode (high electron energy upper than 140 eV) and O_2- and O- in the post discharge (toff low electron energy)[2,3].

We have obtained transparent polycrystalline NiO thin films by fast-HIPIMS with lower crystallization than in DC. We have proved that the preferential direction growth of the structure depends on the oxygen content and on the fast-HIPIMS frequency.

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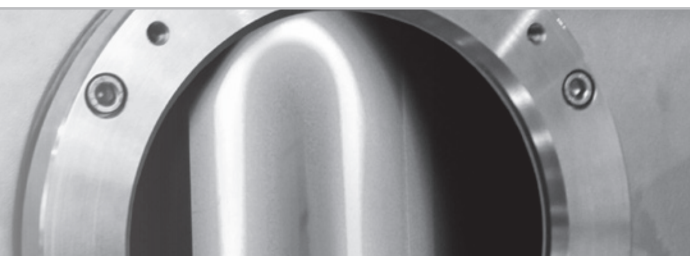


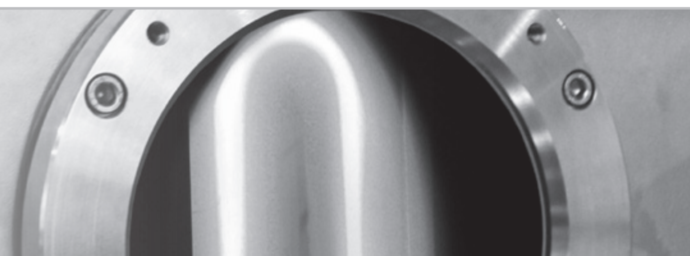
Ice-free windows and other applications with the HIPIMS technology

V. Sittinger*, F. Horstmann, W. Boentoro, W. Werner, B. Szyszka, G. Bräuer

Fraunhofer Institute for Surface Engineering and Thin Films IST, Bienroder Weg 54E, 38108 Braunschweig, Germany

***corresponding author**
volker.sittinger@ist.fraunhofer.de

Functional coatings on weather exposed surfaces produce a huge interest in the architectural glazing and automotive industry. Since these coatings are exposed to atmospheric conditions and natural environment e.g. dust, dirt or salt, their resistance to mechanical abrasion and their chemical stability has to be ensured to guarantee their function over lifetime. The need of a cost-effective process for curved and toughened glass necessitates the use of float glass and a heat treatable and bendable coating. Well insulated glass units (UG < 1.0 W/m²K) as well as windshield on cars mist up at a clear night to a greater or lesser extent depending on the glazing concept and its mounting angle. Mist and water condensation on glass surfaces can disturb the view. A Low-E coating at the outer glass surface such as transparent conductive oxides (TCO) can almost entirely prevent water condensation on its surface.
At Fraunhofer IST a TCO coating has been developed for exterior position based on a high power impulse magnetron sputtering (HIPIMS) applied at flat and three dimensional substrates. Structural analysis has been carried out for films with different deposition parameters before and after heat treatment. Fields of application for this coating are low emissivity coatings, photovoltaics, heat mirrors or panel heaters as well as touch panels and displays.
Film properties have been studied by optical spectroscopy (UV/VIS), atomic force microscopy (AFM) and x-ray diffraction (XRD) measurements. Mechanical stability has been tested with taber-, scratch-, and sand trickling tests. The impacts have been observed with light and scanning electron microscopy as well as haze measurements. Furthermore, ITO films were evaluated in respect to lifetime stability as well as coat and bend applicability.



POSTER Time-resolved laser-induced fluorescence diagnositcs in the HIPIMS plasma

N. Britun[1], M. Palmucci[1], S. Konstantinidis[1], R. Snyders[1,2]

[1] Chimie des Interactions Plasma Surface (ChIPS), CIRMAP, Université de Mons, Place du Parc 20, B-7000 Mons, Belgium

[2] Materia Nova Research Center, 1 Avenue Copernic, B-7000 Mons, Belgium

We present our recent results on laser-induced fluorescence (LIF) plasma diagnostics[1] of a high-power impulse magnetron sputtering (HIPIMS) discharge. The discharge was operating at 1 kHz of the repetition frequency with the pulse duration of 10 µs. The plasma characterization was performed during the sputtering of a 10 cm diameter Ti target in Ar, at 5 and 20 mTorr of the working pressure. The time behaviour of the relative Ti and Ti+ ground state densities as well as their velocity distribution functions (VDF) were studied during the plasma off-time. The measurements were performed at two working pressures, and at three distances from the target (15, 45, 70 mm) uniformly covering the space between the target and the virtual substrate position. It was found that the full width at half maximum (FWHM) of the VDFs of sputtered species in the direction parallel to the target surface demonstrates a faster-than-an-exponential decay during the plasma off-time having always a spectral shape close to the Lorentzian one. Based on these facts a strong non-thermalization as well as strong influence of the collisional broadening on the plasma spectral lines are suggested. The thermalization trends for Ti neutrals and ions were found to be similar, however, the FWHMs of the Ti+ VDFs appear generally narrower. Spatial analysis of the Ti and Ti+ densities show a slight density increase at the intermediate distance from the target which correlates with the previous measurements[2], yet being unclear. The measured Ti+ density shows the presence of two time-resolved peaks, one of which was attributed to the thermalized ions in the discharge, which is in a good agreement with the previous results[3]. In the plasma on-time, where LIF signal was found to be undetectable, time-resolved optical emission spectroscopy of Ti, Ti+, Ar and Ar+ was applied in order to complete the measurements.

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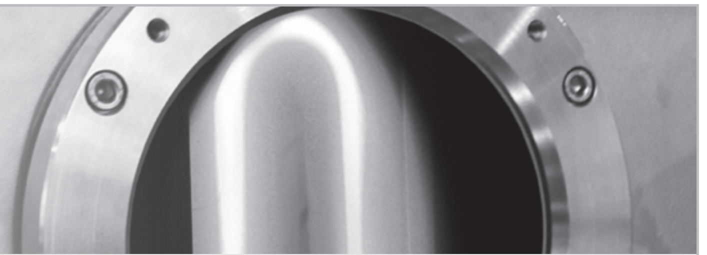


POSTER The distribution of Hall drifts in a HIPIMS discharge

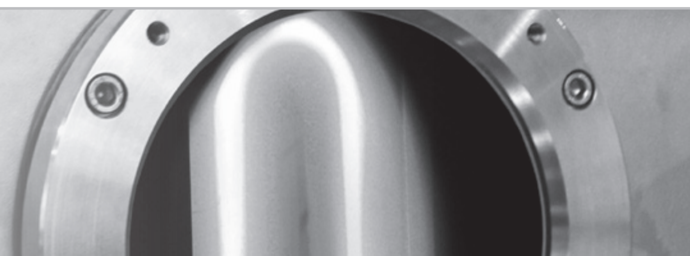
J. Bradley[1]*, A. Mishra[1], P. Kelly[2]

[1] Department of Electrical Engineering and Electronics,
University of Liverpool,
Brownlow Hill, Liverpool L69 3GJ, UK

[2] Surface Engineering Group, Dalton Research Institute,
John Dalton Building, Manchester Metropolitan
University, Chester Street, Manchester, MI 5GD, UK

***Corresponding Author**
E-Mail: j.w.bradley@liv.ac.uk

Combing spatially-resolved measurements of electric and magnetic fields, the distribution of E x B electron drifts have been determined at different times in a HIPIMS discharge pulsed at 100 Hz. The magnetron was operated with at Ti target and at 650 W power. The electric field was obtained from time-resolved emissive probe measurements of the plasma potential during plasma operation while the magnetic field vectors were found from analytical modelling. In the early part of the discharge pulse, large axial and radial electric fields (> 5 kVm^{-1}) close to the target give rise to strong azimuthal electron drifts (velocities > 3 × 105 ms^{-1}) in a broad channel above the racetrack. The peak velocities are about 2 cm from the target, and the distribution is somewhat wider than that observed in DC operation. Later in the pulse (of width 100 µs), at the time of maximum discharge current, the electric fields across the pre-sheath region adjacent to the target reduce and the Hall drift channel moves away from the target revealing a peak in velocity (about 1 × 105 ms^{-1}) some 5 cm into the bulk plasma. During this phase of the pulse, the distribution of Hall electron drifts is more consistent with that observed in magnetrons operated in DC conditions.



POSTER Comparison of residual stress and thermal conductivity of AlN thin films deposited by HIPIMS and DC reactive magnetron sputtering

K. Ait Aissa*, B. Belkerk, A. Achour, Y. Scudeller, L. Le Brizoual, P. Y. Jouan and M. A. Djouadi

Institut des Matériaux Jean Rouxel (UMR 6502), 2 rue de la Houssinière, BP 32229, 44322 Nantes, Cedex 3, France

***Corresponding author**
E-Mail: keltouma.ait-aissa@cnrs-imn.fr
phone: 00 33 (0)2 40 37 39 68

Aluminium nitride (AlN) thin films have attracted great interest thanks to their outstanding physical properties. In general, AlN is a material of high hardness, thermal conductivity, oxydation resistance and chemical inertness. Therefore, AlN thin films have been widely used for electronic applications mainly packaging[1].
AlN thin films can be synthesized by using several techniques[2-4]. Nevertheless, in order to deposit AlN at low temperature, DC and HIPIMS-reactive magnetron sputtering techniques have been used[5, 6].
In the present work, AlN thin films were deposited on Si (100) substrates by DC and HIPIMS reactive magnetron sputtering of an Al target (purity: 99.995 %) in an Ar/N_2 gas mixture for a total pressure of 3 mtorr and a discharge power of 150 W in DC mode and 300 W in HIPIMS. Preliminary results will be presented on the advantage and drawbacks of the use of HIPIMS vs. DC deposition technique in terms of the variation of the film preferential orientation, crystalline quality (FWHM), residual stresses as a function of: 1) thickness, 2) N_2 content in the discharge and 3) temperature. The crystalline and residual stress of AlN thin films were investigated using X-ray diffraction and film-substrate curvature method. Moreover, thermal conductivity measurements have been conducted using electrical and optical methods[7, 8]. The thermal conductivity for films deposited by DC and HIPIMS will be presented and discussed regarding the film and interface quality.

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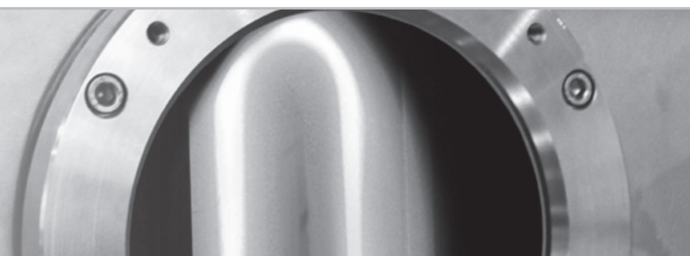


POSTER Investigation of HIPIMS in a reactive atmosphere discharge with Oxygen content by energy resolved mass spectrometry

Y. Aranda Gonzalvo[1]*, A. P. Ehiasarian[2]

[1] Plasma & Surface Division, Hiden Analytical Ltd., 420 Europa Boulevard, Warrington, WA5 7UN, UK

[2] HIPIMS Technology Centre, Materials and Engineering Institute, Sheffield Hallam University, Howard Street, Sheffield, S1 1WB, UK

*corresponding author
E-Mail: gonzalvo@hiden.co.uk
Phone: +44 1925 445225 | Fax: +44 1925 416518

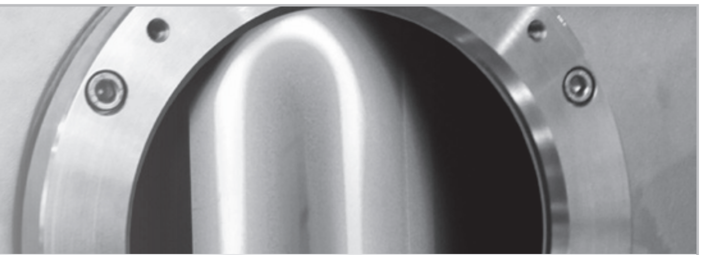
HIPIMS (High Power Impulse Magnetron Sputtering) is a PVD technique for the deposition of high-quality thin films. It allows a high power density to be applied at the cathode, yielding a higher degree of plasma ionization than currently possible in conventional magnetron sputtering process. Due to a low duty cycle, high power density is accumulated into the active part of the pulse. Recently, HIPIMS has been used for deposition of oxides in particular for TiO_2 and TiO_xN_y. It is of great interest to study the effects of the oxygen content in the deposition flux observed in the HIPIMS discharge and its influence on the film microstructure.

In this study, the operation of HIPIMS in an atmosphere of working gases of Ar and N_2 with 20 % O_2 content and a Ti target was investigated. The plasma was operated at a pressure of 1-1.7 Pa and an Ar:Air partial pressure ratio ranging from 45:1 to 45:10 was used to operate at the metallic-to-poisoned point. The peak current was varied from 3 to 10 A with a pulse duration of 200 µs. The frequency was adjusted between 100 and 400 Hz to maintain a constant average power of 0.6 kW. Time-resolved measurements of the plasma parameters near the substrate were carried out using energy resolved mass-spectrometry 170 mm from the target. The main aims of this study were to investigate the influence of the gas metal ion ratio and plasma composition on the film microstructure.

Mass spectrometry measurements showed that the reactive HIPIMS discharge produced a deposition flux with a significantly increased content of ionised film-forming species, such as Ti^{1+}, Ti^{2+}, N^{1+}, O^{1+}, TiO^{1+} and TiO^{21+}. Increasing the air content in the discharge resulted in an enhanced activation of the oxide species, TiO^{1+} and TiO^{21+}, and a reduction in the atomic ion N^{1+}, Ti^{1+} and Ti^{2+}. Ions with energies up to 80 eV were detected during the pulse with reducing energy in the pulse-off times in the metallic mode. The ion energy decreased considerably when the discharge was operated in the oxide mode.

In the mass spectrum of the negative ions only O^- and O^{2-} species were observed. The detailed study of the energy distribution of O^- and O^{2-} shows high-energy ions formed at the cathode with energies proportional to the full cathode voltage. The ion energy distribution function shows that in the transition from metallic to the oxide mode there is about 100 V drop of the voltage on the oxide surface layer on the cathode; this corresponds well with the measured cathode voltage.

The effects of the oxygen content in the deposition flux observed in the HIPIMS discharge on the film microstructure are discussed.



POSTER On the gas rarefaction transient in the beginning of a long HIPIMS pulse

C. Huo[1], N. Brenning[1]*, D. Lundin[2], M. A. Raadu[1], A. Anders[3]

[1] Division of Space and Plasma Physics, School of Electrical Engineering, Royal Institute of Technology, SE-100 44 Stockholm, Sweden

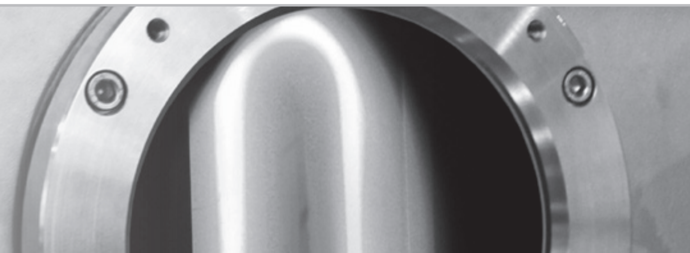
[2] Plasma & Coatings Physics Division, IFM-Materials Physics, Linköping University, SE-581 83 Linköping, Sweden

[3] Lawrence Berkeley National Laboratory, 1 Cyclotron Road, California 94720, USA

***Corresponding author**
Nils Brenning: nils.brenning@ee.kth.se
phone:+46-8-7907688 | fax: +46-8-24 54 31

The interplay between the internal collisional, the cross-boundary convection, and the target sputtering and secondary electron emission mechanisms in a HIPIMS discharge have been investigated using the Ionization Region Model (IRM).

The model is bench-marked against data from a sputtering magnetron with a square voltage pulse in Ar, with an Al target. The model reproduces an initial high-current transient, as well as a later plateau value of constant lower current. The peak discharge current is found to occur just before maximum gas rarefaction, while the full transient length is close to the time it takes to establish a balance between Ar ionization losses and diffusional refill from the surrounding volume. However, not only gas rarefaction but also a significant degree of self sputtering is established during the transient, and influences the nature of the discharge. The physics involved, the evolution of plasma density and electron temperature, and the relative importances of gas rarefaction and metal mixing, are illustrated by model runs tailored to highlight the involved mechanisms.



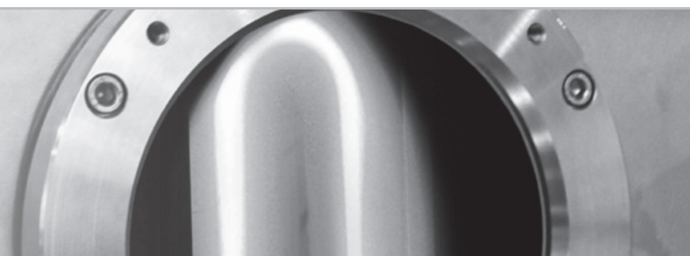
POSTER Advanced control and monitoring circuits create new possibilities for the HIPIMS technology

P. Ozimek, W. Glazek*, A. Klimczak, P. Lach, P. Różański

HUETTINGER Electronic Sp. z o.o., Marecka 47,
05-220 Zielonka, Poland

***corresponding Author**
Wojciech.Glazek@pl.huettinger.com

HUETTINGER incorporated powerful DSP, FPGA and fast data acquisition devices into the control circuits of power supplies dedicated for the HIPIMS technology. This opened new opportunities for HIPIMS research as well as industrial HIPIMS applications. Fully digital control and monitoring, together with a fast data acquisition system, will also bring more flexibility into programming of the power supply's behavior and help realize new ideas.



POSTER Comparison of tantalum nitride thin films deposited by DC Pulsed, HIPIMS and MPP techniques for protective coatings

L. Mendizabal*, U. Ruiz de Gopegui, R. Bayón, X. Fernández, C. Zubizarreta, U. Eletxigerra, J. Barriga

TEKNIKER-IK4 RESEARCH INSTITUTE, Eibar, Spain

***corresponding author**
lmendizabal@tekniker.es

Thin films of transition metal nitrides are widely used as protective coatings due to their high hardness and mechanical strength, corrosion and wear resistance, and chemical inertness. Within them, very little work has been reported on tantalum nitride (TaN). Nevertheless, it shows superior properties on biomedical[1] and tribological[2, 3] applications where properties mentioned above are required. Coatings deposited by the newly developed PVD techniques, such as high power pulsed magnetron sputtering (HIPIMS) and modulated power pulsed (MPP) magnetron sputtering, have revealed outstanding wear and corrosion behavior[4-7]. In the present work, TaN coatings deposited by DC Pulsed, HIPIMS and MPP have been carried out and compared under the same process parameters (pressure, Ar/N_2 gas flow ratio, average target power). The aim of the work is to analyze the improvements achieved by the new techniques as well as the study of the viability of TaN as protective coating.

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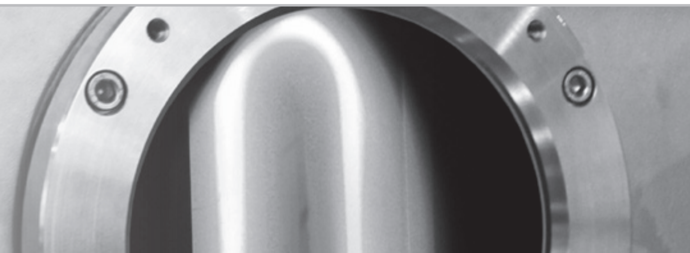
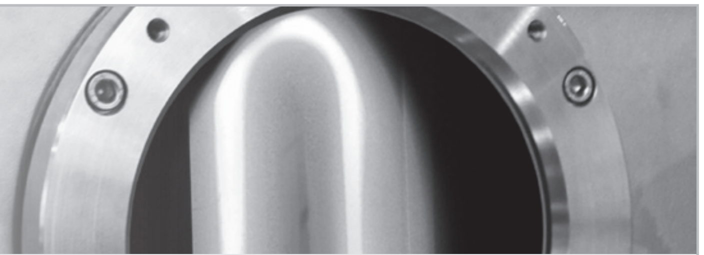


POSTER High power magnetron discharge on graphite target

S. Krassnitzer
OC Oerlikon Balzers AG , Li-9496 Balzers

Hydrogen free diamond like carbon is a very promising thin film material to achieve very hard, wear resistant coatings. Widely used technologies are PECVD and arc evaporation. PECVD is not capable of producing hydrogen free layers, while arc evaporation has the drawback of incorporated macro particles in the film. High Power Sputtering (HIPIMS) of graphite should combine the advantages of the former technologies resulting in hydrogen free layers without incorporation of macro particles. Unfortunately carbon has a very low sputter yield and a very low secondary electron emission coefficient. On top of these comes the high threshold energy for carbon ionization. Neither these conditions are very helpful to easily achieve a magnetron discharge at high current density, nor it´s not likely to get a prominent amount of the sputtered carbon to be ionized. In this work, we show the voltage-current characteristics of a magnetron discharge of graphite, as a function of discharge voltage and the influence of reactive gas.
We found that reasonable current density is only achieved at high discharge voltage above 1400 V. The discharge characteristic is very sensitive on reactive gas, like nitrogen, acetylene or residual gas from outgassing chamber walls. The findings are completed by an estimation of the carbon ionization.

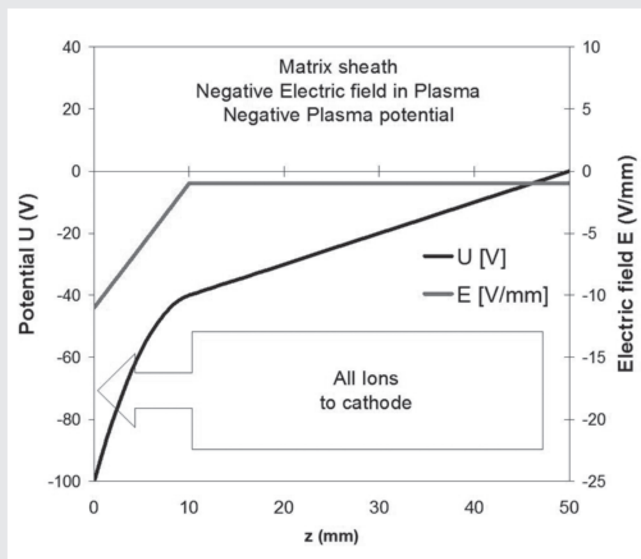


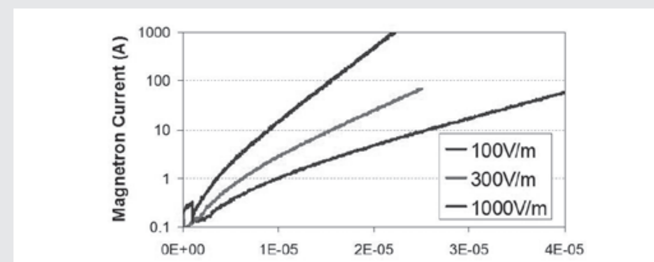
POSTER Use of test electron simulation for analysis of HIPIMS pulse shape based on electric field in presheath and plasma

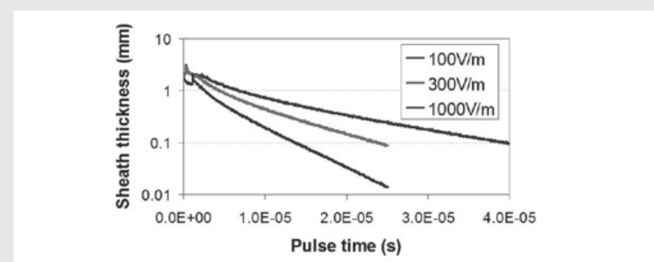
S. Kadlec

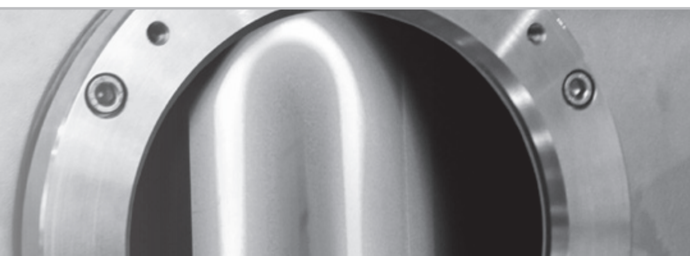
HVM Plasma, Na Hutmance 2, 158 00 Praha 5
Czech Republic
Phone +420 251087125 | Fax +420 251612400
E-Mail: stanislav.kadlec@hvm.cz

Magnetron discharges are difficult to simulate by self-consistent simulation (such as PIC-MC) due to high demand on computing power for simulation of realistic discharge power. The situation of High Power Impulse Magnetron Sputtering (HIPIMS) is even more difficult, due to high power densities in pulse, reaching order of magnitude $1kW/cm^2$.
This work used test-electron simulation that is not self-consistent but has less limitation regarding high current and power densities. The model uses electrons and Ar ions with variable computing weight, moving in 3D magnetic field and one-dimensional electric field. The space-charge within in the sheath is computed from discharge voltage and current density by the Child-Langmuir law or using matrix sheath model. Electrons emitted from the cathode collide with Ar gas and ionize; the ion current density is recorded from delayed ion impact on the cathode. A magnetron with 300 mm diameter in 2Pa Ar has been used for simulation, with approximately 1A DC current at 350 V. Dynamics of discharge current growth from 200 mA to about 100 A after applying 600 V cathode potential has been studied. It has been shown that the electric field in the plasma is quite important for the discharge dynamics. The ionization occurs mainly out of the sheath in the presheath and plasma region at medium or high current densities.
When the electric field is positive, positive ions are accelerated from the target towards the substrate and walls. However, such ions are not used for sustaining the discharge. In case of negative electric field the ions are accelerated towards the cathode. Their acceleration is proportional to the field value, therefore the time delay decreases approximately with square root of the increasing electric field.
The current grows over time nearly exponentially, with more rapid growth at sheath thickness of 1mm or more, when more ions are created in the sheath. A decade of growth from 1 to 10 A discharge current takes about 5 µs at 10 V/cm electric field, whereas 5 µs are needed at 10 V/cm.
The simulation results can be compared with experimental data, especially the pulse shapes and pulse delays. It can also help to understand variation of pulse starting times with pulse frequency, due to various initial plasma density.









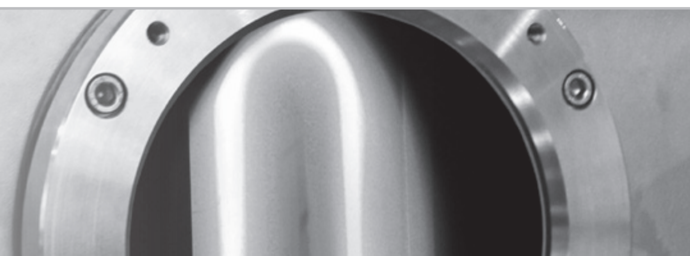
POSTER Microstructure, Oxidation and Tribological Properties of TiAlCN / VCN Coatings Deposited by Reactive HIPIMS

G. Kamath, A. P. Ehiasarian and P. E. Hovsepian

Nanotechnology Centre for PVD Research, Materials and Engineering Research Institute,
Sheffield Hallam University, S1 1WB, United Kingdom

2.5 µm thick TiAlCN / VCN coating was deposited in industrial size 1000×4 Hauzer Techno Coating machine equipped with HIPIMS power supplies manufactured by Huettinger Electronic Sp. z o.o. Prior to the coating deposition the substrate surface was pretreated by bombardment with V+ ions generated in a HIPIMS discharge. A 300 nm thick TiAlN base layer was deposited by operating one magnetron in HIPIMS mode in Ar+N_2 atmosphere. TiAlCN / VCN was deposited in mixed Ar+N_2+CH_4 reactive atmosphere utilising two opposing magnetrons furnished with TiAl and V targets operated in HIPIMS mode.

XTEM analysis of the as deposited coating revealed the formation of three zones with different nanostructures across the film thickness. In the initial stages of deposition a TiAlCN / VCN nanoscale multilayer structure was formed (first zone) followed by a nanocomposite structure comprising TiAlCN / VCN crystalline grains surrounded by a carbon rich tissue phase (second zone). The grain size of the crystalline phase gradually decreased with thickness from diameter of 10 – 15 nm to complete dissolution in the third zone where Me- carbon with XRD amorphous structure was formed. Raman spectra with UV excitation (λ = 325 nm) taken from the top zone 3 of the coating revealed the graphitic nature of the coating showing clearly the G and D peaks at 1577 cm^{-1} and 1396 cm^{-1} respectively with IG / ID ratio of about 0.94. This unique three zone nanostructure is believed to develop due to the progression of the target poisoning effect during the HIPIMS deposition.
In Daimler Benz Rockwell C indentation adhesion test, TiAlCN / VCN showed adhesion class 1. C ratch test critical load values were Lc = 50 N. Pin-on-disc tests revealed the temperature dependence of the coefficient of friction decreasing from µ = 0.6 at room temperature to µ = 0.4 at elevated temperature 650 °C. The wear coefficient showed the opposite trend increasing by two orders of magnitude from 4.2 × 10 – 15 m^3 / N / m at room temperature to 1.2 × 10 – 13 m^3 / N / m at 650 °C which was attributed to the progression of the tribo-oxidation processes. In dynamic oxidation conditions, thermogravimetric analysis determined the temperature of the onset of rapid oxidation to be 800 °C. Compared to UBM deposited TiAlCN / VCN coatings, the oxide mass gain of the HIPIMS coatings was less than 50 %.



POSTER Nanocomposite coatings consisting of noble metal nano-clusters embedded into a dielectric matrix by reactive HIPIMS

N. M. Figueiredo[1], J. C. Oliveira[1]*, T. Kubart[2], A. Cavaleiro[1]

[1] SEG-CEMUC – Department of Mechanical Engineering, University of Coimbra, Rua Luís Reis dos Santos, 3030 - 788 Coimbra, Portugal

[2] Solid State Electronics, The Ångström Laboratory, Uppsala University, Box 534, 751 21 Uppsala, Sweden

***Corresponding author**
joao.oliveira@dem.uc.pt
Phone: +351-239-790745 | Fax: +351-239-790701

The aim of the present research was to synthesize nano-composite coatings with noble metal nanoclusters dispersed or embedded in/into a dielectric matrix (Al_2O_3 or TiO_2) by reactive High Power Impulse Magnetron Sputtering (HIPIMS). This deposition technique allows a better control of the nanoclusters formation and thus eliminating the need of thermal annealing common for DC sputtered coatings. Three power supply configurations were used in this study, high HIPIMS (self-sputtering), low HIPIMS (typical Ar+ type discharge) and pulsed DC, for depositing layers of noble metal thin enough to originate island-typed morphologies of the metal at the surface/embedded of/ into the dielectric matrix. In order to work under the High-HIPIMS mode a pulse length of 300 µs was chosen since longer pulses allowed the discharge to evolve to the metal-plasma discharge. At a voltage of 710 V and higher, the sputtered metal started to strongly affect the discharge and the sustained self-sputtering regime was achieved. For the low HIPIMS mode a shorter pulse was used (pulse length of 50 µs) in order to take advantage of the initial current peak associated with Ar rarefaction. The three deposition modes allowed different size distributions and inter-particles spacing. The size of the Au nanoclusters varied from 4 – 15 nm and they presented different aspect ratios.

All the Ag samples showed strong colors, attributable to the presence of well-defined SPR absorption bands. Moreover, these colors were variable according to the thickness of the metal layer and also to the different sputtering modes, suggesting that the samples should contain clusters with homogeneous shapes and narrow size distributions.

List of authors

Author	Affiliation
B. Abraham	Zond Inc / Zpulser LLC **| USA**
G. Abrasonis	Division of Nanostructures (FWIO), Institute of Ion Beam Physics and Materials Research, Helmholtz-Zentrum Dresden-Rossendorf e. V. **| DE**
A. Achour	Institut des Matériaux Jean Rouxel, Université de Nantes **| FR**
M. Aiempanakit	Plasma and Coatings Physics Division, IFM-Materials Physics, Linköping University **| SE**
A. Aijaz	Plasma and Coatings Physics Division, IFM-Materials Physics, Linköping University **| SE**
K. Ait Aissa	Institut des Matériaux Jean Rouxel, Université de Nantes **| FR**
A. Anders	Lawrence Berkeley National Laboratory | **USA**
J. Andersson	Solid State Electronics, The Angstrom laboratory, Uppsala University **| SE**
Y. Aranda Gonzalvo	Plasma & Surface Division, Hiden Analytical Ltd. **| UK**
N. Argiolas	Università di Padova, Dipartimento di Fisica **| IT**
M. Audronis	Gencoa Ltd **| UK**
A. Balhamri	Chimie des Interactions Plasma Surface (ChIPS), CIRMAP, Université de Mons **| BE** Laboratoire Rayonnement & Matière: Equipe d'Optique Appliquée et Transfert d'Energie (LOPATE) Université Hassan 1er, Faculté des Sciences et Techniques de Settat **| MA**
M. Balzer	FEM – Research institute for precious metals and metals chemistry **| DE**
R. Bandorf	Fraunhofer-Institute for Surface Engineering and Thin Films IST **| DE**
J. Barriga	TEKNIKER-IK4 RESEARCH INSTITUTE **| ES**
R. Bayón	TEKNIKER-IK4 RESEARCH INSTITUTE **| ES**
M. Bazzan	Università di Padova, Dipartimento di Fisica **| IT**
B. Belkerk	Institut des Matériaux Jean Rouxel, Université de Nantes **| FR**
V. Bellido-Gonzalez	Gencoa Ltd. **| UK**
M. Bilek	School of Physics, A28, University of Sydney **| AU**
W. Boentoro	Fraunhofer-Institute for Surface Engineering and Thin Films IST **| DE**
R. Bogdanowicz	University of Greifswald, Institute of Physics **| DE**
M. Böke	Institute of Experimental Physics II, Ruhr-University Bochum **| DE**
S. Bolz	CemeCon AG **| DE**
J. Bradley	Department of Electrical Engineering and Electronics, University of Liverpool **| UK**
G. Bräuer	Fraunhofer-Institute for Surface Engineering and Thin Films IST **| DE**
N. Brenning	Division of Space and Plasma Physics, School of Electrical Engineering, Royal Institute of Technology **| SE**
N. Britun	Chimie des Interactions Plasma Surface (ChIPS), CIRMAP, Université de Mons **| BE**
L. Brohan	Institut des Matériaux Jean Rouxel, Université de Nantes **| FR**
S. Bruns	Fraunhofer-Institute for Surface Engineering and Thin Films IST **| DE**
M. Čada	Academy of Sciences of the Czech Republic, Institute of Physics **| CZ**
A. Campiche	Hauzer Techno Coating BV **| NL**
L. Cattin	Institut des Matériaux Jean Rouxel, Université de Nantes **| FR**
A. Cavaleiro	SEG-CEMUC – Department of Mechanical Engineering, University of Coimbra **| PT**
P. Chapon	HORIBA Jobin Yvon **| FR**
R. Chistyakov	Zond Inc / Zpulser LLC **| USA**
M. Colasuonno	Associazione CIVEN **| IT**
P. Cormier	GREMI, Université d'Orléans **| FR**
C. Costin	Alexandru Ioan Cuza University, Faculty of Physics **| RO**
T. de los Arcos	Institute of Experimental Physics II, Ruhr-University Bochum **| DE**
M. Djouadi	Institut des Matériaux Jean Rouxel, Université de Nantes **| FR**
H. Do	University of Greifswald, Institute of Physics **| DE**
S. Drache	University of Greifswald, Institute of Physics **| DE**
R. Dusart	GREMI, Université d'Orléans **| FR**
A. Ehiasarian	HIPIMS Technology Centre, Materials and Engineering Institute, Sheffield Hallam University **| UK**
U. Eletxigerra	TEKNIKER-IK4 RESEARCH INSTITUTE **| ES**
G. Erkens	Sulzer Metaplas **| DE**
M. Fenker	FEM – Research institute for precious metals and metals chemistry **| DE**
X. Fernández	TEKNIKER-IK4 RESEARCH INSTITUTE **| ES**
A. Ferrec	Institut des Matériaux Jean Rouxel, Université de Nantes **| FR**
F. Fietzke	Fraunhofer-Institute for Electron Beam and Plasma Technology FEP **| DE**
N. Figueiredo	Department of Mechanical Engineering, University of Coimbra **| PT**
P Frach	Fraunhofer-Institute for Electron Beam and Plasma Technology FEP **| DE**
M. Ganciu	National Institute for Laser, Plasma and Radiation Physics, Plasma Department **| RO**
H. Gerdes	Fraunhofer-Institute for Surface Engineering and Thin Films IST **| DE**
W. Glazek	HUETTINGER Electronic Sp. z o.o. **| PL**
C. Gottfried	Fraunhofer-Institute for Electron Beam and Plasma Technology FEP **| DE**
G. Greczynski	Department of Physics, Linköping University **| SE**
A. Hecimovic	Institute of Experimental Physics II, Ruhr-University Bochum **| DE**
R. Heller	Institute of Ion Beam Physics and Materials Research, Helmholtz-Zentrum Dresden-Rossendorf e. V. **| DE**
U. Helmersson	Plasma and Coatings Physics Division, IFM-Materials Physics, Linköping University **| SE**
A. Hendriks	Hauzer Techno Coating BV **| NL**
R. Hippler	University of Greifswald, Institute of Physics **| DE**
F. Horstmann	Fraunhofer-Institute for Surface Engineering and Thin Films IST **| DE**
P. Hovsepian	Nanotechnology Centre for PVD Research, Materials and Engineering Research Institute, Sheffield Hallam University **| UK**
Z. Hubička	Academy of Sciences of the Czech Republic, Institute of Physics **| CZ**
L. Hultman	Department of Physics, Linköping University **| SE**
C. Huo	Division of Space and Plasma Physics, School of Electrical Engineering, Royal Institute of Technology **| SE**
L. Jastrabík	Academy of Sciences of the Czech Republic, Institute of Physics **| CZ**
J. Jensen	Department of Physics, Linköping University **| SE**
P. Jouan	Institut des Matériaux Jean Rouxel, Université de Nantes **| FR**
S. Kadlec	HVM Plasma **| CZ**
G. Kamath	Nanotechnology Centre for PVD Research, Materials and Engineering Research Institute, Sheffield Hallam University **| UK**
A. Karpinski	Institut des Matériaux Jean Rouxel, Université de Nantes **| FR**
P. Kelly	Surface Engineering Group, Dalton Research Institute, Manchester Metropolitan University**| UK**
A. Klimczak	HUETTINGER Electronic Sp. z o.o. **| PL**
H. Klostermann	Fraunhofer-Institute for Electron Beam and Plasma Technology FEP **| DE**
Š. Kment	Academy of Sciences of the Czech Republic, Institute of Physics **| CZ**
W. Kölker	CemeCon AG **| DE**
S. Konstantinidis	Chimie des Interactions Plasma Surface (ChIPS), CIRMAP, Université de Mons **| BE**
T. Koranda	Department of Physics, University of West Bohemia **| CZ**
T. Kozak	Department of Physics, University of West Bohemia **| CZ**
S. Krassnitzer	OC Oerlikon Balzers AG **| LI**
T. Krug	Hauzer Techno Coating BV **| NL**
T. Kubart	Solid State Electronics, The Ångström Laboratory, Uppsala University **| SE**
P. Lach	HUETTINGER Electronic Sp. z o.o. **| PL**
J. Lazar	Department of Physics, University of West Bohemia **| CZ**
L. Le Brizoual	Institut des Matériaux Jean Rouxel, Université de Nantes **| FR**
O. Lemmer	CemeCon AG **| DE**
D. Loch	HIPIMS Technology Centre, Sheffield Hallam University **| UK**
D. Lundin	Plasma and Coatings Physics Division, IFM-Materials Physics, Linköping University **| SE**
J. Mahrholz	Fraunhofer-Institute for Surface Engineering and Thin Films IST **| DE**
J. Mathias	GREMI, Université d'Orléans **| FR**
D. McKenzie	School of Physics, A28, University of Sydney **| AU**
R. Mendelsberg	Lawrence Berkeley National Laboratory | **USA**
L. Mendizabal	TEKNIKER-IK4 RESEARCH INSTITUTE | **ES**
A. Mishra	Department of Electrical Engineering and Electronics, University of Liverpool| **UK**
J. Müller	Sulzer Metaplas **| DE**
S. Nakano	National Institute of Advanced Industrial Science and Technology (AIST) **| JP**
R. New	HIPIMS Technology Centre, Sheffield Hallam University **| UK**
D. Nguyen	Institut des Matériaux Jean Rouxel, Université de Nantes **| FR**
H. Ogiso	National Institute of Advanced Industrial Science and Technology (AIST) **| JP**
J. Olejníček	Academy of Sciences of the Czech Republic, Institute of Physics **| CZ**
J. Oliveira	Department of Mechanical Engineering, University of Coimbra **| PT**
N. Ouldhamadouche	Institut des Matériaux Jean Rouxel, Université de Nantes **| FR**
P. Ozimek	HUETTINGER Electronic Sp. z o.o. **| PL**
A. Pajdarova	Department of Physics, University of West Bohemia **| CZ**
M. Palmucci	Chimie des Interactions Plasma Surface (ChIPS), CIRMAP, Université de Mons **| BE**
F. Papa	Hauzer Techno Coating BV **| NL**
A. Patelli	Associazione CIVEN **| IT**
I. Petrov	University of Illinois at Urbana-Champaign **| USA**
G. Popa	Alexandru Ioan Cuza University, Faculty of Physics **| RO**
M. Raadu	Division of Space and Plasma Physics, School of Electrical Engineering, Royal Institute of Technology **| SE**
T. Rasa	Sulzer Metaplas **| DE**
A. Rauch	Lawrence Berkeley National Laboratory **| USA**
J. Rezek	Department of Physics, University of West Bohemia **| CZ**
M. Richard-Plouet	Institut des Matériaux Jean Rouxel, Université de Nantes **| FR**
P. Różański	HUETTINGER Electronic Sp. z o.o. **| PL**
U. Ruiz de Gopegui	TEKNIKER-IK4 RESEARCH INSTITUTE | **ES**
M. Samuelsson	Plasma and Coatings Physics Division, IFM-Materials Physics, Linköping University **| SE**
J. Sanders	Lawrence Berkeley National Laboratory **| USA**
K. Sarakinos	Plasma and Coatings Physics Division, IFM-Materials Physics, Linköping University **| SE**
C. Schiffers	CemeCon AG **| DE**
V. Schulz-von der Gathen	Institute of Experimental Physics II, Ruhr-University Bochum **| DE**
Y. Scudeller	Institut des Matériaux Jean Rouxel, Université de Nantes **| FR**
N. Semmar	GREMI, Université d'Orléans **| FR**
L. Sirghi	Alexandru Ioan Cuza University, Faculty of Physics **| RO**
V. Sittinger	Fraunhofer-Institute for Surface Engineering and Thin Films IST **| DE**
R. Snyders	Chimie des Interactions Plasma Surface (ChIPS), CIRMAP, Université de Mons **| BE** Materia Nova Research Center **| BE**
V. Straňák	Academy of Sciences of the Czech Republic, Institute of Physics **| CZ**
A. Surpi	Associazione CIVEN **| IT**
V. Sushkov	University of Greifswald, Institute of Physics **| DE**
B. Szyszka	Fraunhofer-Institute for Surface Engineering and Thin Films IST **| DE**
A. Thomann	GREMI, Université d'Orléans **| FR**
R. Tietema	Hauzer Techno Coating BV **| NL**
V. Tiron	Alexandru Ioan Cuza University, Faculty of Physics **| RO**
B. Treverrow	School of Physics, A28, University of Sydney **| AU**
M. Vergöhl	Fraunhofer-Institute for Surface Engineering and Thin Films IST **| DE**
J. Vetter	Sulzer Metaplas **| DE**
J. Vlcek	Department of Physics, University of West Bohemia **| CZ**
T. Wallendorf	IfU Diagnostic Systems GmbH **| DE**
A. Weeks-Ross	School of Physics, A28, University of Sydney **| AU**
O. Werner	Fraunhofer-Institute for Surface Engineering and Thin Films IST **| DE**
W. Werner	Fraunhofer-Institute for Surface Engineering and Thin Films IST **| DE**
J. Winter	Institute of Experimental Physics II, Ruhr-University Bochum **| DE**
H. Wulff	University of Greifswald, Institute of Physics **| DE**
K. Yukimura	National Institute of Advanced Industrial Science and Technology (AIST) **| JP**
L. Zottarel	Associazione CIVEN **| IT**
C. Zubizarreta	TEKNIKER-IK4 RESEARCH INSTITUTE **| ES**

BOOK OF ABSTRACTS

2ND INTERNATIONAL CONFERENCE ON FUNDAMENTALS AND APPLICATIONS OF HIPIMS

Local Organiser:

Network of Competence
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Network of Competence
Bienroder Weg 54 E
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