## Hard Coatings and Vapor Deposition Technologies
## Room On Demand - Session B1

### PVD Coatings and Technologies

**B1-1 On Electron Heating and Ion Recycling in the High Power Impulse Magnetron Sputtering Discharge**, 
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In the past it has been assumed that the magnetron sputtering discharge is maintained by the sheath acceleration of secondary electrons emitted from the target, upon ion impact. This is described by the well-known Thornton equation, which in its original form [1] is formulated to give the minimum required voltage to sustain the discharge. However it has been demonstrated recently that Ohmic heating of electrons outside the cathode sheath is roughly of the same order as heating due to acceleration across the sheath in dc magnetron sputtering (dcMS) discharges [2]. Furthermore, for the high power impulse magnetron sputtering (HiPIMS) discharge we find that direct Ohmic heating of the plasma electrons is found to dominate over sheath acceleration by typically an order of magnitude [3]. In HiPIMS discharge a high density plasma is created by applying high power pulses at low frequency and low duty cycle to a magnetron sputtering device. Here we discuss the large discharge currents and the discharge current composition at the target surface in HiPIMS discharges. We discuss the role of self-sputter(SS-) recycling and working gas recycling within the discharge. We find that above a critical current density $J_{crit} \approx 0.2$ A/cm$^2$, a combination of self-sputter recycling and working gas-recycling is the general case [4]. For high self-sputtering yields, the discharges become dominated by SS-recycling, contain only a few energetic secondary electrons. For low self-sputtering yields, the discharges operated above $J_{crit}$ are dominated by working gas recycling, and secondary electrons play a more important role. We explore a discharge with Al target which develops almost pure self-sputter recycling, a discharge with Ti target that exhibits a mix of self-sputter recycling and working gas-recycling [5] and a reactive Ar/$O_2$ gas mixture where working gas-recycling is dominating [6].

[1] J A Thornton, J. Vac. Sci. Technol. 15 (1978) 171

[2] N. Brenning et al., Plasma Sources Sci. Technol. 25 (2016) 065024

[3] C Huo et al., Plasma Sources Sci. Technol. 22 (2013) 045005

[4] N. Brenning et al. Plasma Sources Sci. Technol. 26 125003 (2017)

[5] C. Huo et al., J. Phys. D: Appl. Phys. 50 354003 (2017).

[6] J. T. Gudmundsson et al. Plasma Sources Sci. Technol. 25, 065004 (2016).

**B1-2 Tailoring the Chemical Composition and Microstructure of $Cr_xN$ Deposited by HiPIMS through Duty-cycle Modifications**, 
***Martha Cedeño-Vente (m.cedeno@posgrado.cidesi.edu.mx)***, *G. Mondragón-Rodríguez, N. Camacho, A. Gómez-Ovalle, J. González-Carmona, J. Alvarado-Orozco, D. Espinosa-Arbeláez* , Centro de Ingeniería y Desarrollo Industrial (CIDESI), Mexico

The main challenge in hard coating manufacturing is developing dense, flawless, and smooth surface morphology coatings, which improve the performance of components exposed to severe service conditions. One of the most promising coating techniques to achieve this goal is the High-Power Impulse Magnetron Sputtering (HiPIMS), which produces highly ionized plasmas, resulting in definite improvements on the film microstructure and properties. The application of pulses (50 - 500 µs) during the deposition process attains a high ionization rate in HiPIMS. The pulse modification (*e.g.,* duty-cycle, also known as τ) affects the current density, modifying the stoichiometry and chemical composition of the coating; this indicates that the pulse appropriate selection is a viable and practical alternative for obtaining graded or multilayer metal nitrides at a fixed gas flow, thus tailoring the coating internal stress distribution, microstructure, and mechanical properties.

In this work, the duty-cycle design to modify the chemistry, microstructure, and residual stresses of $Cr_xN$ coatings is presented and discussed. The average current tends to increase from 2.9 to 12 % with the variation of τ during the HiPIMS process. These process conditions lead to a significant increment of the deposition rate and the chromium content in the $Cr_xN$ coating. Various crystalline phases like α-Cr, α-Cr + h-$Cr_2N$, h-$Cr_2N$, and h-$Cr_2N$ + c-CrN were obtained in the film, resulting in graded multilayer systems. Cr-rich samples presented faceted columns with intercolumnar pores and cauliflower-like surface morphology. The growth of the h-$Cr_2N$ phase caused a decrease of the grain sizes, and their morphology changed to pyramidal or stacked pyramids at lower duty-cycles. The transformation of the h-$Cr_2N$ to CrN leads to highly dense columnar microstructures, while the residual stresses of the coating increased with the higher duty-cycle.

***Keywords:*** *HiPIMS, duty-cycle, $Cr_xN$, graded microstructure.*

**B1-3 New Approaches for AlCrN-Based Coatings for High Speed Applications**, 
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The CO2 debate—a global challenge requires the necessity of renewed investments and innovations by manufacturers and suppliers with a strong focus on an increase of sustainability. Especially, the automotive sector (driven by the governmentally regulated CO2 emission limit), in detail the trends for the e-mobility, which has reached higher levels of importance. Consequently, high-performance materials and versatile material combinations with focus on weight reduction and energy-efficient automotive design are replacing conventional materials. The trend to a reduction of the cycle times of the production lines, leads to new challenges in the application of tools, such as higher mechanical and thermal loads exposure during operation.

Thus, the talk will focus on the technical possibilities to overcome these challenges by the appropriate choice of coating design using the example of aluminum-chromium nitride based coatings.

**B1-4 Investigation of the Influence of the Thickness of Nanolayers in Wear-resistant Layers of Ti-TiN-(Ti,Cr,Al)N Coating on Destruction in the Cutting and Wear of Carbide Cutting Tools**, 
***Alexey Vereschaka (dr.a.veres@yandex.ru)***, *S. Grigoriev,* MSTU Stankin, Russian Federation; *N. Sitnikov,* National Research Nuclear University MEPhI, Russian Federation; *J. Bublikov,* Ikti Ran, Russian Federation

The paper presents the results of the investigation into the formation of the nanolayer structure of the Ti-TiN-(Ti,Cr,Al)N coating and its influence on the thickness of coatings, their resistance to fracture in scratch testing, and the wear resistance of coated tools in turning 1045 steel. The structure of the coatings with the nanolayer thicknesses of 302, 160, 70, 53, 38, 24, 16, and 10 nm was studied using scanning electron microscopy (SEM), transmission electron microscopy (TEM), and high-resolution (HR) TEM. It is shown that the grain sizes in the nanolayers decrease to certain values with an increase in the thickness of the nanolayers, and then, with a further decrease in the nanolayer thickness, the grain sizes of the nanolayer grow as the interlayer interfaces cease to produce a restraining effect on the growth of the grains. The study found that the nanolayer thickness influenced the wear of carbide cutting tools and the pattern of fracture for the Ti-TiN-(Ti,Cr,Al)N coatings.

**B1-5 INVITED TALK: Industrial Scale ta-C Coating Using Laser Arc Technology**, 
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First reports about a Laser-initiated vacuum arc date back to 1976 /1/. Laser ignition has many advantages particularly for pulsed high current vacuum arcs when spatial and temporal control at high pulse repetition rates for millions of pulses is required. This holds especially for the carbon arc which deviates markedly from metal arcs with respect to arc movement, charge state and degree of ionization and particle generation. The carbon arc is preferentially operated at pulsed arc currents in the kA range and pulse lengths of some 100 µs. The Carbon Laser-Arc has been investigated and applied to deposition of highly tetrahedral amorphous carbon films (ta-C) since the 1990th/2/. Only in recent years the Carbon Laser-Arc was introduced in mass production for coating of automotive powertrain components as well as for other tribological applications and cutting tools. In this paper we discuss different aspects of upscaling ta-C Laser-Arc coating systems in order to increase the throughput and the total amount of deposited carbon per batch. The importance of process stability for long coating runs is discussed. We also report on deviating film properties that have been observed on films deposited at intermittent high-rate deposition of ta-C.

/1/ J.E. Hirshfield, Laser-initiated vacuum arc for heavy ion sources. IEEE Transact. Nucl. Sci. 23, 1006-1007 (1976)

/2/ H.-J. Scheibe et al, Laser-arc: a new method for preparation of diamond-like carbon films. Surf. Coat. Technol. 47, 455-464 (1991).

**B1-7 Key Importance of the Controlled Reactive HiPIMS for Low-temperature Preparation of Tunable Oxynitrides and Thermochromic Oxides***, Jaroslav Vlček (vlcek@kfy.zcu.cz), J. Houška,* University of West Bohemia, Czech Republic

Reactive high-power impulse magnetron sputtering (HiPIMS) with a feedback pulsed reactive gas (oxygen and nitrogen) flow control and to-substrate reactive gas injection into a high-density plasma in front of the sputtered metal target was used for a low-temperature deposition of highly optically transparent Al-O-N films (substrate temperature of 120 °C) and thermochromic $VO_2$-based films (substrate temperature of 330 °C) onto unbiased substrates.

A modified version of HiPIMS, called Deep Oscillation Magnetron Sputtering, was used to produce high-quality Al-O-N films with a gradually changed elemental composition (from $Al_2O_3$ in AlN), structure and properties. We give the basic principles of this controlled deposition, maximizing the degree of dissociation of both $O_2$ and $N_2$ molecules in a discharge plasma, which leads to a replacement of very different reactivities of the $O_2$ and $N_2$ molecules with metal atoms on the surface of growing films by similar (high) reactivities of atomic O and N.

We developed a low-temperature scalable deposition technique for high-performance durable thermochromic $ZrO_2/V_{0.982}W_{0.018}O_2/ZrO_2$ coatings. The $V_{0.982}W_{0.018}O_2$ layers were deposited by controlled HiPIMS of V target, combined with a simultaneous pulsed DC magnetron sputtering of W target (doping of $VO_2$ by W to reduce the transition temperature to 20-21 °C), in an argon-oxygen gas mixture. The effective pulsed oxygen flow control of the reactive HiPIMS deposition makes it possible to utilize the enhanced energies of the ions bombarding the growing $V_{0.982}W_{0.018}O_2$ layers for the support of the crystallization of the thermochromic phase in them at the low substrate surface temperature of 330 °C and without any substrate bias voltage. We present the basic principles of this controlled deposition.

**B1-8 Monitoring Tantalum Nitride Thin Films Structure by Reactive HiPIMS Magnetron Sputtering: From Microstructure to Properties***, Angeline Poulon-Quintin (angeline.poulon@icmcb.cnrs.fr), A. Achille,* ICMCB-CNRS, France*; D. Michau,* Univ. Bordeaux, ICMCB, France*; M. Cavarroc,* Safran Tech, France

Tantalum nitride thin films were deposited onto steel substrates using Reactive High-Power Impulse Magnetron Sputtering (HiPIMS) allowing reaching a high ionization degree of the sputtered metallic material thanks to high power density applied to the target during few tens of microseconds pulse. The influence of target power density, $N_2$ partial pressure, total gas ($Ar + N_2$) pressure and target-to-substrate distance on film crystalline structure is reported. The structures obtained were investigated by X-ray diffraction and transmission electron microscopy. Cubic metastable phase or hexagonal stable phase can be successfully isolated in single phase continuous layer. The TaN crystalline phase obtained depends strongly on processing parameters especially pulse parameters. It is well known that TaN hexagonal single-phase continuous layer is difficult to isolate using conventional reactive magnetron sputtering. Our previous study, based on RF magnetron sputtering, has shown TaN hexagonal structure formation to be enhanced in growth conditions promoting adatoms mobility on the substrate surface. With HiPIMS, TaN hexagonal phase layer is much more difficult to obtain due to the increase number of process parameters to select, the specific composition and the energy of the plasma created. Comparison of mechanisms involved during the stabilization of each TaN structure depending on the process is presented as well as characterization of microstructure, and properties (mechanical, electrical and optical).

**B1-9 INVITED TALK: Multilayer nano-composite Oxidation-resistant Coatings for Accident-tolerant Nuclear Fuel Cladding using Reactive HiPIMS with Positive Kick and Precision Ion Energy Control***, Brian Jurczyk (bjurczyk@starfireindustries.com), R. Stubbers, I. Shchelkanov, T. Houlahan,* Starfire Industries LLC, USA **INVITED**

In the wake of the Fukushima nuclear accident in Japan there is worldwide pressure to improve the accident tolerance of fuels used in light water reactors. A near-term pathway is to deposit a thin protective coating directly on existing Zr-alloy fuel cladding trading coating properties (i.e. chemical resistance, wear resistance, fracture toughness, radiation damage), impact on neutron economy and thermal hydraulics, manufacturing feasibility and implementation readiness, per cladding unit, CapEx and operations costs, regulatory acceptance and quality assurance protocols, and applicability for both pressurized-water and boiling-water conditions. In this study we evaluate a high-throughput fabrication method for nanolayered Cr-based corrosion-resistant and fracture-resistant coatings using a high-power impulse magnetron sputtering innovation—namely the IMPULSE® + Positive Kick™. Ultra-fast IMPULSE® switching achieves high instantaneous plasma densities during HIPIMS discharge pulse for easy control over self-sputtering, ionization fraction and reactive gas management. The adjustable Positive Kick™ quickly reverses the polarity on the sputter target to accelerate metal ions to the substrate—increasing delivered ion fraction and rate for higher efficiency. Precision ion energy control results in fully dense films across a wider range of the Thornton diagram controlling film stress and morphology. Metals and ceramics are precision deposited with excellent adhesion, graded composite nanostructure and conformal layering for radiation hardness, thermal shock- and oxidation-resistance. ~10µm Cr-based coatings were deposited via IMPULSE® + Positive Kick™ on 9.5-mm diameter Zr-alloy cladding with Ar and $N_2$ gas pressure (0.5-5Pa), cathode power density (0.1-2kW/cm$^2$), main pulse width (5-100µs), Positive Kick™ voltage (+0-600V), kick delay & width (0-100µs), and repetition rates up to 10kHz. Utility of in-situ surface cleaning via the Positive Kick™ is also demonstrated for adhesion. Samples were characterized pre- and post-testing using mechanical testing, optical and scanning electron microscopy (EDS, EBSD) and x-ray diffraction. Thin-film microstructure was evaluated using SEM, EDS, EBSD and FIB. Corrosion tests were performed in an autoclave using boronated and lithiated water at 360°C at 18.7MPa over sequential time periods for weight gain and spallation/delamination inspection. Manufacturability estimates for volume Zr-alloy coating using a patent-pending inverted cylindrical magnetron configuration optimized for conformal HiPIMS deposition is presented.

**B1-12 Multifunctional Coatings with Antifouling Properties***, Jose Castro (jodcastroca@unal.edu.co), I. Carvalho, M. Henriques, S. Carvalho,* University of Minho, Portugal

Additive manufacturing (AM) is a hot topic nowadays, having a first order in importance in research trends, improving existent technologies and carrying them further. AM can be applied to all family types materials: metals, polymers, ceramics and compounds. Among abovementioned, ceramics have a huge importance and application in our current technologies. Their capability to maintain functional properties for long time periods combined with the easiness to process and abundance of raw materials make them a fundamental part of mankind development. Within this type of materials, one of the most commonly used nowadays is stoneware. This material has a wide range of uses, from everyday usage such as kitchenware and pottery to high tech applications such as pipelines, which in some cases are affected by biofouling. Some ceramics are not able to prevent the formation of biofouling formation which can affect their finish and appearance. Trying to solve this issue, TiN and Ag:TiN with oxygen addition coatings in 3D printed stoneware, were presented as multifunctional solution, in order to extend the performance of base material, offering a variety in an aesthetical point of view and adding antibacterial properties. This study performed the aforementioned films by reactive direct current (DC) magnetron sputtering. Films obtained were characterized physical, chemical and morphologically, as well as their color variation, roughness, wettability, antibacterial and antibiofouling resistance. The results revealed that the Ag doped coatings (with or without oxygen addition) had an enhanced multifunctionality compared with control samples (without Ag). The Ag nanoparticles addition created a surface with antibacterial and antibiofouling, in order to resist outdoors and aqueous environments, making these films able to be applied in architectural pieces as sculptures or other decorative parts, maintaining their properties with good aesthetical properties.

**B1-13 How to Deposit a Porous Thin Film by Magnetron Sputtering ?***, Diederik Depla (Diederik.Depla@ugent.be), R. De Doncker,* Ghent University, Belgium

Without additional efforts, thin films deposited by magnetron sputtering are dense due to the bombardment by sputtered atoms and reflected neutrals. To overcome this intrinsic feature of magnetron sputtering, several routes have been explored to deposit a porous thin film, and still to benefit from the advantages of magnetron sputtering.

Increasing the deposition pressure and/or tilting the substrate belongs to the most common approaches, but these strategies are plagued by technical issues such as enhanced arcing, a low deposition rate, and limited scalability.

Another strategy is based on the deposition of a mixture of materials, or an alloy which is chemically and/or physically treated to remove one of the constituents. Dealloying by applying a heat treatment and subsequently electrochemical etching is one example that belongs to this group of methods. High temperature and/or (electro)chemical treatments limit the substrate choice, and are often environmentally harmful.

This paper suggests an alternative approach based on the sputter deposition of a powder mixture of a metal with NaCl [1]. The deposited layer is simply treated with water to dissolve the deposited salt, leaving a porous metal layer on the substrate. The thin films are deposited from a powder target composed of NaCl and metal powder. Due to the low thermal conductivity of the target, the target gets hot. As a result, the salt is sublimed while the metal is sputtered from the target. The film composition, and therefore the porosity, is controlled by the applied target power.

This one-source approach without the use of film annealing or aggressive chemicals overcomes the major obstacles of other synthesis routes without compromising the benefits of magnetron sputtering.

[1] Sputter deposition of porous thin films from metal/NaCl powder targets, R. Dedoncker, H. Ryckaert, D. Depla, Appl. Phys. Lett. 115(2019) 041601doi:10.1016/10.1063/1.51128225

**B1-14 Evolution of the Microstructure of Sputter Deposited TaAlON Thin Films with Increasing Oxygen Partial Pressure***, Nina Schalk*

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Recently, quaternary oxynitrides of transition metals and aluminum have attracted increasing interest due to their tunable properties. Within the present work, a series of TaAl(O)N films was sputter deposited using constant nitrogen and varying oxygen partial pressures. The films were grown from single element Ta and Al targets. The deposition parameters were adjusted to obtain a Ta/Al atomic ratio of ~50/50 for the oxygen-free film and were held constant for the following depositions, with the exception of the increasing oxygen partial pressure and compensatory decreasing argon partial pressure. Elastic recoil detection analysis revealed oxygen contents of up to ~26 at.%, while the nitrogen content decreased from ~47 at.% in the oxygen-free film to ~35 at.% in the film with the highest oxygen content, resulting in a significant decrease of the metal/non-metal ratio with increasing oxygen partial pressure. The micro- and bonding structures of the films were investigated by X-ray diffraction, X-ray photoelectron spectroscopy, Raman spectroscopy and transmission electron microscopy. All films exhibited a dominating face centered cubic TaN-based structure with indications for an additional nanocrystalline phase fraction, which increases with increasing oxygen partial pressure. In addition, the mechanical properties were evaluated by nanoindentation, yielding a decreasing hardness and elastic modulus with increasing oxygen content.

**B1-15 Towards Knowledge-based Design of Multi-element Target Materials***, Mehran Golizadeh*

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The increasing demand for multi-element thin films and coatings for multifunctional purposes has pushed the target and cathode material industry to produce multi-element products. Nowadays, alloyed and composite targets are commonly employed in various physical vapor deposition technologies including magnetron sputtering and cathodic arc deposition. The targets, which serve as cathodes in the respective discharges, are exposed to the discharge plasma during the deposition process, which alters their surface properties such as phase and chemical composition. The surface modifications are particularly severe for cathodic arc deposition since the cathode spots impose countless melting-solidification cycles on the target material near the surface, leading to the formation of a network of craters and a several 10 µm thick modified layer. The formation mechanisms and properties of the modified layer, semi-empirically quantified by the cohesive energy of the constituent phases, influence the charge states and kinetic energies of the ions and, hence, the film growth conditions and coating properties.

In a first step, a Mo/Al multilayer cathode was designed to reveal information about the heat-affected zone below the craters as well as the evolution of material intermixing as the sequential cathode spot events take place. The modified layer is formed by micro-sized displacements of the cathode material during crater formation. In addition, the material intermixing predominantly occurs in liquid state while the mechanisms based on solid-state diffusion play an insignificant role due to the sharp temperature gradient (shallow heat-affected zone) below craters and very high cooling rates [1]. As a next step, the microstructure and phase composition of the modified layers of Al-Cr composite cathodes with varying grain sizes were studied. The results from high-resolution analysis techniques revealed that metastable phases, including quasicrystalline phases, are formed during the solidification of arc craters and, hence, are the constituting phases of the modified layers. The average cooling rate in these rapid solidification processes was estimated to be in the order of $10^6$ K/s. Accordingly, to optimize the plasma properties for film and coating depositions it is necessary to consider non-equilibrium phases of the alloy system as they might be present on the modified surface. This means that the target's or cathode's microstructure and constituent phases need to be designed to enable the formation of phases with optimized cohesive energy during the rapid solidification of arc craters.

1. Golizadeh, M., et al., J. Appl. Phys., 2020. 127(11).

**B1-16 INVITED TALK: Coating Design and Mechanical Properties of Multicomponrnt AlTi(X)N Hard Coatings***, Yin-Yu Chang*

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Due to economical demands to further increase the efficiency of production processes, it is essential to exploit the full potential of wear resistant hard coatings. TiN and AlTiN-based coatings are widely used as protective material for cutting tools, molds, and mechanical components in mechanical industries. Low chemical reactivity of these hard coatings with workpiece materials protects against sticking and thus reduces the adhesive wear. The most widespread wear resistant coatings are those with the following chemical formula Ti-X-(N,C and B) (X = Al, Cr, C, Si, and B etc.) which have proven to have excellent properties for industrial applications in the cutting, forming and stamping fields.

In this study, the coating design, mechanical property, high temperature oxidation behavior and cutting performance of multicomponent and multilayered AlTi(X)N coatings, which X= Cr, Si and B etc., will be discussed. These high performance coatings can be deposited by using cathodic-arc deposition with arc cathodes or unbalanced magnetron sputtering. Various cathode targets, such as Ti, Cr, TiAl, TiAlSi, CrAlSi, and AlSi, are used for the deposition. The microstructure of the as-deposited and high temperature annealed coatings was characterized by field emission scanning electron microscope (FESEM), high resolution transmission electron microscope (HRTEM) and X-ray diffraction (XRD) using Bragg-Brentano and glancing angle parallel beam geometries. The mechanical properties including hardness and elastic modulus of the coatings were analyzed by a nanoindenter with Berkovich indenter tip.

Depending on the coating design, the deposited AlTi(X)N coatings showed B1-NaCl crystal structure and have multiple orientations of (111), (200), and (220). The nanohardness, which measured by nanoindentation, of these coatings possessed hardness higher than 30 GPa, depending on the gradient and multilayered structures. The high temperature oxidation test showed the oxidation rate during annealing depends on film composition and microstructure. The oxide layer formed on the AlTiSiN coatings consists of large $TiO_2$ and AlTiSiN grains at the oxide-coating interface, followed by a layer of protective $Al_2O_3$ in the near-surface region. Interestingly, after oxidation, the AlTiBN coating contained an oxide layer composed of nanocrystalline $Al_2O_3$ and $TiO_2$. No crystallite growth or phase transformation occurred in the unoxidized AlTiBN coating part after oxidation. The gradient, multilayered, and nanocomposite AlTi(X)N show significantly improvement of the lifespan of cutting tools and mechanical parts.

**Keywords**: Hard coating; Mechanical property; AlTiN; Multicomponent; Multilayer

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