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# Plasma and energy flux characterization in high power impulse magnetron sputtering

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Over the course of 25 years since the influential paper by Kouznetsov et al. on high power impulse magnetron sputtering (HiPIMS) [1], numerous studies have demonstrated the significant potential of HiPIMS for thin film deposition. This potential is evident through the enhancement of film quality, specifically in terms of increased density [2] and adhesion [3] along with the diminished requirement for high substrate temperatures [4]. To achieve the optimal deposition process, it is crucial to develop a comprehensive understanding of the plasma-surface interaction on the substrate. This includes, in particular, analyzing the energy flux and its composition.

Measuring the contributions of neutral and ion energy flux onto the substrate provides valuable information about the energy carried by the incoming particles, which greatly affects the film properties. To accomplish this, so-called non-conventional plasma diagnostics such as passive thermal probes (PTP) [5] and a novel combination of PTP with a retarding field analyzer (RFEA) [6] can be employed. These diagnostics have been applied to compare the HiPIMS and DC magnetron sputtering processes with same gas pressure and same average power using a planar copper target. In addition, I-V-characteristics and optical emission spectroscopy (OES) have been used for process control.

The emission spectra reveal e.g. the characteristic gas rarefaction phenomenon in HiPIMS, along with the expected identification of $Cu^+$ ions. The ionized flux fraction is estimated with RFEA measurements since the neutral flux fraction can be measured by repelling incoming charged species by applying a high voltage to the scan grid [6]. In total, the mean energy flux to the substrate is lower in HIPIMS processes. Hence, temperature sensitive substrates are protected. However, it can be inferred from the lower deposition rate in HiPIMS that the incoming particles have higher energy and provide dense film growth.

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# Advancements in Nitrogen-Doped DLC coating for biopolymer moulding: bridging laboratory insights to field applications

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It is known that N-doped DLC coatings can be deposited with various techniques, like PACVD or microwave sources for plasma generation. Experimental data show that Nitrogen doping of a-C:H structure makes the coatings electrical conductive and usually improves their corrosion resistance (based on EIS analysis), while the typical tribological behaviour of Carbon based films is maintained. The addition of dopant elements to amorphous matrix in the form of a-C:H-X modifies surface free energy of such formulation; in this work, the influence of Nitrogen content on this film property is investigated.

Nitrogen doping modifies mechanical properties of the film in the way of its hardness reduction and stress relief as well, resulting in a different fatigue resistance that can be measured by impact testing technique. Non-doped MW DLC coating deposited on HSS polished sample is used as a reference for multiple impact test and then compared with most interesting Nitrogen doped formulations.

Once all the laboratory results were collected and analysed, the most promising N-DLC recipe had been selected to be tested directly on a field test consisting in biopolymer injection moulding. In details, we designed a specific mould for small cups in PLA material with strict aesthetic requirements, resulting in a very demanding application.

Two polymers having distinct viscous flow index were tested with an injection press, giving different results in terms of extraction forces, transparency of the material and localized defects on moulded parts: even in this case the standard non-doped MW DLC coating was considered as reference.

Parallel laboratory analysis were carried out, measuring friction coefficient of a specific PLA against DLCs, deposited with and without Nitrogen addition, by the mean of pin-on-disk reciprocating technique with coated flat samples and PLA moulded balls as counterparts. Another interesting simulation done consists in a traction test designed to measure adhesion force between melted PLA and coated samples.

Output of field tests demonstrate that Nitrogen doped DLC coated mould has required lower extraction forces to release the injected parts.

Finally, this is the only tested solution that guaranteed a consistent production with constant quality parts, resulting a very promising coating solution for biopolymers injection moulding as industrial application as well.

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# Time resolved diagnostics of e-HiPIMS discharge

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HiPIMS (High Power Impulse Magnetron Sputtering) [1] consists in generating a high-power pulsed discharge (some 10-100 times higher than in the DC process) during a very short discharge time (usually less than 1 ms), and a long post-discharge time. The high power enables us to generate a very dense cold plasma close to the target and to achieve vapor ionization rates of a few tens of percent. Increasing the ionization rate considerably improves film quality (denser films [2], better microstructure [3]) and enables deposition on complex substrates.

This HiPIMS regime introduces the duty cycle as a crucial parameter, representing the ratio of discharge to rest times, which impacts the deposition process. To analyze the discharge, it is necessary to perform time-resolved diagnostics to obtain more information during the pulse. Time-resolved mass spectrometry could be used [4]. It showed that ions do not arrive at the same time: gas ions are present initially, and metal ions are generated later.

To modify these different times, a 6-stage laboratory voltage generator (e-HiPIMS) has been made [5]. With this generator, presented for first time at HiPIMS 2021 [6], we can add further pulses to see the differences between HiPIMS processes. Figure 1 shows the different processes used. We employed a range of techniques including time-resolved mass spectrometry, optical emission spectroscopy, X-ray diffraction, and scanning electron microscopy to gain a clear understanding of how the coatings behave under various conditions. (See figure 1).

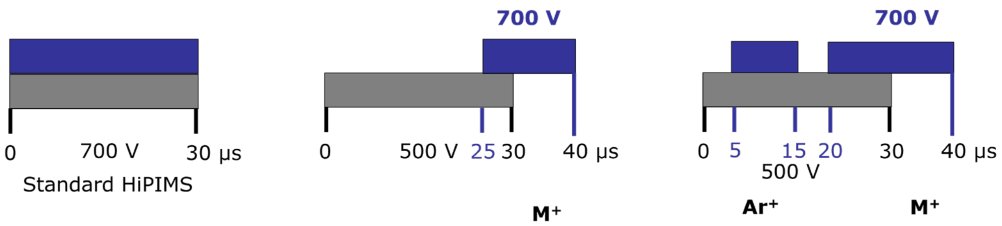


Figure: Different types of process used with e-HiPIMS to modify the ion distribution function of $Ar^+$ and metallic ion ($M^+$)

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# Plasmonic arrays prepared at room temperature by HIPIMS deposition of titanium nitride

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Nanostructured plasmonic arrays are of interest for catalysis and sensing applications. Transition metal nitrides (TMNs) such as titanium nitride (TiN) are plasmonic materials with increased mechanical stability, thermal stability, and spectral tunability when compared to the noble metals gold and silver and as such have emerged as promising alternative materials for tailored applications [1,2]. TiN has potential applications for plasmonic and optoelectronic applications in the visible and near-IR spectral ranges.

However, the deposition of TMNs with optical properties suitable for plasmonic applications typically requires high temperatures (> 800 °C). Device fabrication methods are therefore restricted to those compatible with such high deposition temperatures, often at the expense of scalability or added process complexity. To successfully develop TMN-based optoelectronic devices it is therefore desirable to deposit high quality thin films at lower, CMOS compatible temperatures (< 400 °C) [3].

In this work, we use High-Power Impulse Magnetron Sputtering (HIPIMS) to deposit plasmonic TiN thin films at room temperature. The optical, structural, and morphological properties of the thin films are fully characterised via spectroscopic ellipsometry, XRD, and AFM. Furthermore, we present the fabrication of plasmonic TiN nano-features and investigate their optical properties and plasmonic performance.

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# Stable hybrid HiPIMS/RF sputtering process on a single magnetron for arc-free deposition of compact oxide films

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Thin, insulating coatings are required for electronics, sensors and medical technology. Most of them are deposited by reactive magnetron sputtering and involve an RF or MF excitation of the plasma (radio/mid frequency). However, this often results in sub-stoichiometric layers with process-induced, but undesired residual porosity. With HiPIMS (high power impulse magnetron sputtering), significant advantages over conventional sputtering processes can be achieved, such as the production of coatings with high adhesion and almost bulk density. However, the deposition rates are lower when compared to an RF or MF process with the same average power. In addition, process stabilization is not trivial due to high peak currents and short pulse durations. Instabilities are induced by arcing between insulating areas on the target, leading to droplet formation, which significantly reduces the achievable film quality [2]. To overcome these difficulties, we have for the first time investigated the combination of an RF and HiPIMS excitation in a single magnetron.

Therefore, a HiPIMS generator from Melec company was combined with a RF-Generator from Caesar and connected to a single magnetron. To avoid back reflections, a special RF-Filter from Aurion was used. $Al_2O_3$ layers were deposited in a hybrid RF/HiPIMS process using a metallic Al target and $O_2$ as reactive gas, with variations in power and pulse parameters.

A stable reactive hybrid RF/HiPIMS process on a single magnetron, with higher process stability when compared to a simple HiPIMS process, has been demonstrated for the deposition of $Al_2O_3$ layers. The number of arcing events could be significantly reduced. A higher deposition rate with higher nano hardness of the deposited coatings could be achieved [5].

A proof of principle for a combination of RF and HiPIMS excitation in one source has been established and opens up a new route for the arc-free deposition of $Al_2O_3$ and other oxidic layers. Further investigations will include the influence and optimization of pulse parameters as well as the relationship of average HiPIMS and RF power. For a pulsed superposition of RF and HiPIMS, further developments of ultrafast impedance matching techniques are also necessary.

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# Tuning the film properties on insulating substrates using multi-pulse bipolar HiPIMS

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The introduction of high-power impulse magnetron sputtering (HiPIMS) has brought further possibilities for controlling the deposition process due to the high fraction of ionized species and broadened ion energy distribution function. Recently, bipolar HiPIMS, where the main negative voltage pulse is followed by a positive one, has been suggested to be used instead of a substrate bias voltage. However, this approach is often limited when the plasma-substrate potential difference is lost due to the charging of the insulating substrate. This work proposes to utilize multi-pulse bipolar HiPIMS to facilitate energy delivery to films on insulating substrates.

The experiments were performed using a magnetron with a Ti target driven by an in-house built HiPIMS power supply controlled by a two-channel waveform generator. This configuration allowed us to investigate discharge conditions including standard HiPIMS, standard bipolar HiPIMS, and several non-standard variations of multi-pulse bipolar HiPIMS. Ti films were deposited on insulating substrates, and their properties were analyzed. In addition, ion mass- and energy-spectroscopy analyses were performed to understand the observed phenomena.

The carried out experiments have revealed that the proposed approach provides more possibilities to control the film properties on insulating substrates as demonstrated in Fig. 1. Here, the compressive stress of the films increases according to the pulse configuration used. We provide an explanation of the observed behavior based on a longer total time during which the desired positive plasma-substrate potential difference is kept during the multi-pulse HiPIMS. In addition, the carried-out spectroscopy has shown that the multi-pulse approach yields a broader peak with an elevated high-energy tail.

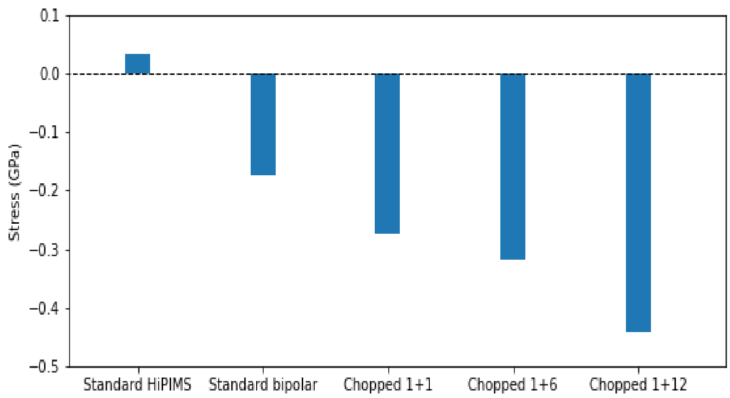


Figure: Stress in the Ti films prepared on insulating substrates using standard HiPIMS, standard bipolar HiPIMS and different variations of multi-pulse bipolar HiPIMS.

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# Impact of substrate bias voltage on characteristics of HiPIMS deposited TiAlSiN and its performance in near-dry machining of austenitic stainless steel

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In the current investigation, the substrate bias voltage exerts a presumably predominant and significant influence on the microstructural morphology and mechanical properties of HiPIMS-deposited coatings. This investigation delves into the intricacies of a quaternary coating, namely TiAlSiN, deposited on cemented carbide (WC-10 wt.% Co) substrates and solid carbide end-mills using the HiPIMS process. Utilizing a pair of TiAl60 and TiSi34 targets, four different substrate biases were applied: – 60, – 90, – 120, and – 150 *V*, to explore variations in coating characteristics. In each case, the substrate bias was applied in pulsed mode with a 40 *µs* offset to the cathode pulse.

The impact of Ti, Al, and Si atomic percentages on the microstructure and mechanical properties of the coatings was investigated using various techniques, including X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), field emission scanning electron microscopy (FESEM), high-resolution transmission electron microscopy (HRTEM), 3D-surface profiling, nano-indentation, Rockwell indentation-adhesion, and scratch test. The coating's thickness and deposition rate were estimated from the cross-section morphology. A novel approach, Fermi-edge referencing, has been used to analyse the XPS spectra. The HRTEM and XPS results suggested that the TiAlN phase likely existed in an amorphous phase of $Si_3N_4$ in TiAlSiN coatings. Observations also revealed an increase in both cathode current (from 87 to 98 *A*) and bias current (from 19 to 23 *A*) with an increase in substrate bias voltage. Elevated bias voltage resulted in finer surface morphology, and the cross-section morphology became notably denser. The Ti content (at%) exhibited a range of 28.35 to 30.71 with increasing bias voltage. The coating achieved its maximum hardness at – 150 *V*, recording 42.95 *GPa*. The adhesion level, determined by Rockwell indentation, was consistently classified as *HF1* in all deposition cases. However, scratch test results indicated a load range between 154 to 161 *N* for complete coating delamination. Primary failure mechanisms during scratching were identified as spallation and edge-chipping in the TiAlSiN coating.

The coated solid carbide end-mills were subjected to machining of austenitic stainless steel under wet and near-dry machining environments. Cutting fluid flow rate was used only 250 *ml/hr* under near-dry, whereas under wet conditions, the flow rate was set to be 25 *l/hr*. The formation of the built-up-edge was completely arrested; however, a thin built-up-layer was identified on the exposed carbide under both cutting environments. The result indicates a significant improvement in flank wear and crater wear of 50 *µm* and 33 µm, respectively, after machining 20 *m* under near-dry cutting conditions using the TiAlSiN coated end-mill deposited at a substrate bias of –150 *V*. This improvement suggests the effectiveness of the coating and deposition conditions in enhancing tool performance and wear resistance during machining operations.

**Keywords:** HiPIMS; TiAlSiN; Bias-offset; HRTEM; XPS; Near-dry; Flank wear.

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# Effect of magnetic field on reverse discharge ignition in bipolar HiPIMS

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A phenomenon associated with bipolar HiPIMS is the occurrence of the so-called reverse discharge (RD) when a sufficiently long positive pulse is applied. It arises due to the formation of a double-layer structure. A recent study [1] suggests that the main cause of the charge double-layer formation and RD maintenance is the secondary electron emission triggered by $Ar^+$ ions colliding on grounded surfaces and the magnetic mirror effect of the magnetron's magnetic field. The magnetic field prevents electrons in the bulk plasma from reaching the magnetron, causing them to accumulate behind the magnetic trap. At certain conditions, the plasma potential behind the magnetic trap drops, and the double-layer is formed. Then, electrons accelerate within this structure, causing the discharge to ignite. The magnetic field configuration is assumed to be a key factor affecting the plasma potential distribution and the RD ignition during the positive pulse, but its effect has not yet been systematically investigated.

This study reveals the crucial impact of magnetic field configuration on bipolar HiPIMS discharge by analyzing floating potentials and optical emission spectroscopy (OES) imaging. Previous studies [1, 2] showed that the changes in the plasma potential associated with RD ignition are also manifested in the time evolution of the floating potential. This study confirms that the RD ignition can be easily detected by monitoring the floating potential by a wire probe. Furthermore, the sensitivity of the RD to the placement of external electrodes near the discharge plasma is identified.

The experiments are performed using a magnetron with a 100 mm Ti target in a stainless-steel vacuum chamber at 1 Pa. The discharge was operated at 500 W average power with a 100 µs negative pulse, followed by a 500 µs positive pulse (100 V amplitude after a 10 µs delay). Magnetic field adjustments were made by moving the inner and outer magnets independently. Wire probes measured floating potential at different distances, and an em-ICCD camera captured Ar atoms' light emission during positive pulses.

In a more balanced magnetic field configuration, the drop in floating potential, indicating RD ignition, occurs earlier. Conversely, in an unbalanced configuration, electrons can reach the target more easily, causing a delayed onset of the RD or even its absence. Consequently, the RD can be more easily avoided using a more unbalanced magnetic field and shorter positive pulses.

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# Ultrafast hot electron diffusion dynamics in titanium nitride

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Titanium nitride (TiN) is a ceramic material with strong broadband absorption and tunable stoichiometry [1]. Especially, it has high free carrier density which make it exhibits optical properties like gold in the visible and ear-infrared regimes [2]. Additionally, titanium nitride shows enhanced hot electron harvesting relative to gold [3, 4] and have long-lived hot carriers [5]. However, the physical mechanism of those phenomenon and intrinsic properties of hot carriers in TiN are poorly understood. In this work, we investigated the hot electron diffusion dynamics by imaging the spatial-temporal hot-electron diffusion in TiN through ultrafast scanning thermo-modulation microscopy [6]. We revealed two distinct diffusion regime, a fast initial diffusion process within the first several tens picoseconds, and the following 200 times slower diffusion at longer time scale. We attribute these two processes are the pure hot electron diffusion and phonon-limited thermal diffusion. Both of these two processes are faster than those in gold [6]. Next, we are going to develop a comprehensive model of the diffusion dynamics to further confirm our conclusion.

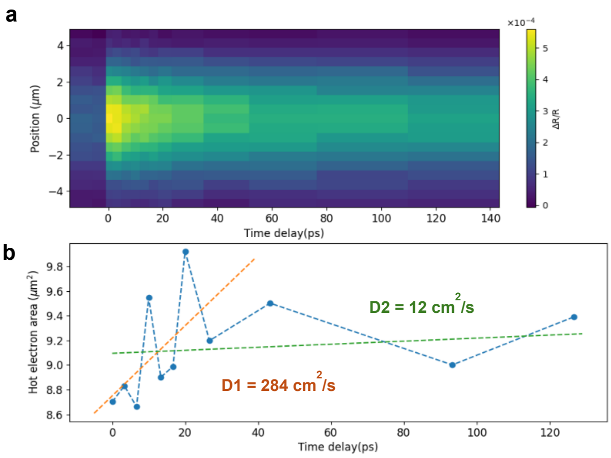


**Figure:** Hot carrier diffusion dynamics in TiN film. (a) Spatiotemporal dynamics of the time resolved reflection signal $\Delta R/R$. The pump wavelength is 840 nm, the probe wavelength is 1200 nm. (b) Squared width $\Delta R/R$ evolution of the profile, extracted by Gaussian fitting to the spatial profile at each time delay.

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# HIPIMS – Fascinating technology to make next steps in decorative and functional applications

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The PVD market is moving fast towards new application fields. To realize these new applications, different PVD coating techniques are used and HIPIMS is among the most fascinating ever since it’s discover. Over the last 25 years several developments are being carried out on the HIPIMS power supply side such as modification in bipolar mode, pulse shape, pulse length, pulse trains and higher frequency just to name a few. Synchronization of cathodes, as well as with HIPIMS based bias has lead PVD coating solution to new track.

Beside the already known performance gain on HIPIMS coated cutting tools - HIPIMS has shown it’s potential for several other applications. We will show and explain the potential of making different colors with the use of HIPIMS. Different advantages on several aspects of HIPIMS for decorative applications on 3D products will complete the picture. For components - HIPIMS is good tool to enhance wear and corrosion properties of existing material system. In our presentation we will show, that especially the combination of HIPIMS with new material systems can even further enhance and widen the potential application areas.

Decorative markets as well as tribological markets are always production cost driven – so coating volume/size matters. To meet these demands, we scaled our HIPIMS developments up, to be able to deposit these coatings on our largest industrial size platform, the Flexicoat 1500, to scarify these market wishes.

We will also give an outlook what could be expected next.

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# Industrial scale high reactive index coatings – control and monitoring on the example of $TiO_2$ and SiAlN

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Reactive HIPIMS deposition is known for it's potential of advanced coating properties. Monitoring or active control of the reactive processes demands suitable hardware for the processes. While $TiO_2$ is mainly attractive due to the high refractive index of more than 2.7 at 550 nm, SiN or SiAlN is often used as scratch resistant top coat, e. g. in architectural glazing.

This contribution will focus on active monitoring and control for better understanding of fundamental aspects of reactive HIPIMS and realization of reliable HIPIMS deposition processes. For $TiO_2$ as model system the hysteresis behavior and control aspects are discussed. In the case of SiAlN the focus is on improved scratch resistance. Therefore, a reactive HIPIMS process was evaluated resulting in advanced coating properties compared to the DC-reference process.

Main attention was devoted to the investigation of a relationship between the nitrogen content in the process gas and the pulse length on the films and their mechanical and optical properties. Both can be indirect measured by Haze. Optical properties were measured by UV-VIS-NIR.

It was found that for low nitrogen content (<21%) the films were less transparent and absorb at wavelengths below 500 nm, leading to higher Haze values. In turn the films with higher nitrogen content of 33% or more were highly transparent. Results of a pulse length variation showed that the pulse length have an impact on the Haze of the Si-Al-N films. Pulse times of 50 µs showed the best results with Haze values of 0.46.

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## Reactive and non-reactive deposition of tungsten carbide by HIPIMS

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Tungsten carbide (WC) is a widely used hard compound for cutting tool substrates known as hard metal or cemented carbide. Usage of WC for protective coatings is less explored especially when it comes to the HIPIMS PVD method. In this study, we deposited WC by reactive sputtering using acetylene gas and also by co-sputtering with pure graphite and tungsten. For the latter we applied a combinatorial approach with an upper and lower target segment so that we could vary chemical composition or the W:C ratio vertically in one process. We also varied substrate bias voltage from 100 to 200 V. The resulting coating thickness was kept around 1 µm which proved to be sufficient for nano-hardness and XRD measurements. High plastic hardnesses of more than 35 GPa were obtained for tungsten rich coatings due to formation of the $W_2C$ phase. Carbon contents of above 50 atomic % quickly led to a drop in hardness as graphite is accumulating in the coating. Compared to constant bias (DC) a pulsed bias generally led to higher hardness. However, pulsed bias voltages above 175 V led to a degradation and loss in hardness. A time delay between cathode and bias pulse had only a minor effect on hardness and modulus. Wear testing by blasting with abrasive grit and machining of aluminium alloy showed an improved wear resistance of the hardest $WC_X$ coatings compared to standard TiAlN hard coatings.

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# Powder metallurgical target manufacturing – targets design challenges in case of depositing high entropy alloy coatings

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Nowadays not only tools for machining but also components for different industries are coated by utilizing physical vapor deposition methods. University research and industrial development both work to introduce optimized coating deposition methods but also to implement new material compositions.

One of these new materials are the so-called high entropy alloys and their coatings. Despite the ongoing scientifical debate about the nature and nomenclature of these types of coatings, target materials are required to fulfill to deposit such coatings.

As new coatings and processes become more common, the target manufacturers must support the development and deliver suitable solutions for each requirement.

The targets used for PVD coating applications are produced either by powder or by melting metallurgy processes. Targets manufactured by powder metallurgy are characterized by several advantageous properties such as uniform microstructure, high density, as well as homogeneity concerning distribution of chemical elements. The quality of such targets depends on the manufacturing process and for the most part on the quality of the powder ingredients used.

For the development of coatings, the research is focusing on beneficial effects by alloying with selected elements to control the composition of the coating. One of the challenges is to find a suitable technology for production of targets containing all the requested elements on the one side and to consider the impact of the purity of the targets on the whole production chain and the performance of the final product on the other side. On the other hand, the development of high entropy materials requires also new approaches from target manufacturers to serve these ideas and to find ways to develop challenging targets including several powders of different structure, melting points and varying density.

To support the efforts of equipment manufacturers and coating developers, design and technology requirements must be combined to produce targets in appropriate shape and dimensions.

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# Simulating the transport of atoms during HiPIMS deposition of NbC from a compound target

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This presentation focuses on explaining the observed change in the composition of NbC films deposited by HiPIMS from a single compound target for varying pulse-average target power density [1]. Recent diagnostics of the discharge plasma [2] identified the most relevant phenomena affecting the film composition: the ionization and return of sputtered Nb atoms, increased scattering of C above the target and film resputtering by Ar atoms backscattered from the target. But it was difficult to quantify these effects and directly correlate with experiment without a complex numerical simulation.

Therefore, we employ computer simulations based on an in-house DSMC code which enables to simulate the transport of atoms and ions and their mutual collisions in the discharge. The simulation is fully three-dimensional and is constrained by target voltage and current waveforms which ensures good compliance with experimental discharge conditions. It assumes an *a priori* defined plasma potential distribution. This enables to simulate the full duration of HiPIMS pulses at reasonable computational time.

We calculate the degree of ionization of Nb and C above the target, the rates of C and Nb collisions in the discharge plasma and estimate the flux and energy of backscattered Ar atoms onto the substrate. We correlate these simulation results with the experimentally observed trend in film composition. The sensitivity to input data describing the target sputtering, such as energy and angular distribution of sputtered atoms (calculated by the SDTrimSP code) is as also discussed.

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# Digitalisation of the magnetic driven cathodic arc spot motion – a semi empiric modeling approach

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Tool coating by the cathodic vacuum arc technology is widely used in industry. The highly ionized coating plasma is produced by micron sized arc spots moving around on the cathode surface. This motion is critical for coating quality, cathode utilization and ionisation, that's why the prediction of the spot behaviour by a computer model becomes of growing interest. The basically random spot motion can be steered by magnetic fields.

A mathematical-physical model is introduced that allows for an examination and characterization of the arc spot motion with regard to direction and speed as a function of an imposed magnetic field [1].

This model considers the different components of random walk, retrograde, and Robson drift motion.

Introduced empirical coefficients were determined by corresponding experimental investigations.

The calibrated model describes the arc spot motion in good agreement to the recorded spot tracks and can therefore be applied for an evaluation of different magnetic field configurations.

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# Tempering transformed tough TiAlN: towards a robust implementation on cemented carbide substrates

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With their unique combination of high hardness and thermal stability, transition metal nitrides (TMNs) are the predominant choice for protective tool coatings in metal cutting applications. However, owing to their covalent bond character and comparatively complex crystal structures they are also particularly brittle materials, which ultimately limits their capabilities under the abrupt loading of cutting forces.

In search of measures to mitigate this limitation, it was found that the fracture toughness of certain metastable PVD nitrides may be significantly increased via thermal annealing treatments at temperatures >> 800°C [1,2]. Unfortunately, in the prevalent case of cemented carbide tools, such elevated temperatures also induce detrimental reactions between the coating and substrate that have so far prevented any robust utilization of annealing treatments.

The present contribution revisits the potential of pre-aged (Ti,Al)N coatings, complementing previous studies with micro-pillar splitting and machining tests. It furthermore addresses the challenge of substrate-coating-reactions by evaluating potential interdiffusion barriers based on low-kV EDX and microscale shear testing.

While interactions between substrate and coating are highlighted as a major challenge to any implementation of annealing-treated TMNs, it is demonstrated that purpose-built coating laminates may enable tool life improvements of more than 100% with respect to corresponding as-deposited references or other state of the art PVD coatings.

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# Reverse pulse strategies for silicon dioxide thin films deposition by High Power Impulse Magnetron Sputtering

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Transparent oxide layers such as silicon dioxide play a vital role in a wide variety of applications in modern architecture from photovoltaics to low e glazing as well as in displays and optical sensors. Utilising High Power Impulse Magnetron Sputtering (HIPIMS) it is possible to deposit high density and high hardness layers. In this study the effect of utilising dual-magnetron bipulse mode and reverse voltage on the coating properties and arc likelihood were examined.

Utilisation of the constant current mode in the pulses prevented process instabilities usually linked to current runaway in reactive processes.

$SiO_x$ was deposited from pure silicon targets in a reactive $Ar\text{-}O_2$ atmosphere. Peak power density was 0.5A/cm$^2$, the pulse duration was up to 20 µs. OES observation of Ar neutral lines (Ar I) showed that plasma persisted to more than 150 µs after the pulse switch off.

Arcing rates were lowest in the bipolar mode of operation and were significantly reduced using reverse pulses due to the discharging of the target surface. In single-target operation, the delay between the end of the pulse and application of the reverse voltage had a strong influence on arcing rates. Applying a reverse voltage after allowing the plasma near the target to decay slowly after switch-off, resulted in inefficient target neutralisation and higher arcing rates. Application of reverse voltage immediately at the end of the pulse utilised high density plasma to neutralise the target and eliminate arcing. SiOx films deposited without additional heating or substrate biasing had a refractive index of 1.43-1.48, similar to bulk glass. A transparency of 97 % and k-value of $10^{-3}$ for 200 nm thick films indicated low defect density achieved at the lowest arc rate. The nanohardness of 1 µm thick films was 10±1 GPa and Young's modulus 77±9 GPa, representing a 10% increase against the glass substrate.

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# The role of the magnetic field design in optimising the performance of Hipims based sputter deposition processes

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The use of the Hipims power mode to create an ionised coating flux from a magnetron sputtering device is just one method available to coat surfaces. Whilst still a relatively small part of the overall sputtering industry, it is experiencing growth and has outperformed other methods in certain areas.

A magnetron based sputtering device has two essential elements to create the coating flux; an applied negative bias to create the target material sputtering effect, and a magnetic confinement to provide trapping of the plasma to create higher density plasmas.

The nature of the magnetic field has a strong influence and works in combination with the chosen power mode. When applying a DC, pulsed DC or RF biasing mode to sputter device, the magnetic field confinement can be adjusted to improve the yields and uniformity of the process.

In the case of a Hipims power mode, the influence of the magnetic field is more fundamentally in creating a more efficient coating process as there is the addition of ionised metal species alongside the electron and the inert gas ions. Additionally, the very high periodic power applied creates a different type of plasma confinement as well as increasing probability of a plasma arc.

In 'managing' and optimising a Hipims based process, the strength and shape of the magnetic field as well as the interaction of the field with anodic surfaces will determine the overall coating rate and quality. It is also the case, that applying Hipims to different target materials requires a different magnetic design if coating rates and quality are to be optimised.

The talk will give practical examples of the effect of the different magnetic fields for a range of Hipims based processes and applications.

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# Sustainable and economical production of high-quality HiPIMS coatings

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Constant improvement of ceramic coatings for cutting tools aiming at best wear resistance under conventional and extreme conditions is driven by the development of new workpiece materials with improved properties. Economical machining of such materials requires ever denser and harder coatings with better adhesion to the tool substrate. In addition to the required coating properties, however, the economical production of these coatings plays a more important role since some time. Shorter coating processes, reduced handling and lower energy consumption are the right keywords to well describe the current situation.

Considering these aspects, high-performance coating technologies, such as HiPIMS, are becoming more and more interesting for the market. Thanks to HiPIMS dense, hard, adhesive, and droplet-free layers can be deposited in highest quality with high energy efficiency at high deposition rates. Furthermore, well-chosen HiPIMS pulse parameters combined with an appropriate bias synchronization can avoid high residual stress of coatings for sharp edged cutting tools.

In our presentation we show that optimization of HiPIMS pulse parameters leads to a significant increase in metal ionization, accompanied by improved coating properties of an (Al,Ti,Si)N layer. The improved coating properties include above all a denser microstructure and a smoother surface, which allows to skip time consuming and energy-intensive post-treatment steps. Brilliant shine and best optical appearance are related with low friction and perfect chip removal during use. This combination of layer properties is a guarantee for a perfect surface finish of the workpiece.

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# Temporal plasma evolution during HIPIMS discharge and its relationship to the pulse time

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The research focuses on examining the temporal dynamics of a HIPIMS discharge with active current control during the pulse time. Experiments were conducted using a 600×200 mm AlSi target in an argon atmosphere. Plasma ignition was initiated by applying high-power impulses of 8 kW average, with voltages reaching up to 1800 V and peak currents up to 600 A. Throughout the tests, the average power remained constant by adjusting the pulse frequency.

Time-resolved optical emission spectral lines, including gas (Ar I, Ar II) and metal (Al I, Al II, Si I, Si II) species, were monitored. The HIPIMS discharge progressed through two distinct phases: the gas region and the metal region, akin to voltage-driven HIPIMS discharge.

The influence of various parameters on the temporal evolution of spectral line intensities was also examined. The ionization wave profile notably shifted with changes in pulse frequency. During extended pulses and off-times, there was a noticeable delay in the evolution of Si I and II emission compared to Al due to differences in sputtering yield. However, at higher frequencies and shorter off-times, ionization of metal species occurred more rapidly without delay compared to gas species. The spectral lines of excited species developed in phase with each other throughout the entire period, which can be attributed to a higher density of residual plasma facilitating the ignition of subsequent HIPIMS pulses.

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## Bactericidal efficacy and surface morphology of nanopatterned TiN films deposited by High Power Impulse Magnetron Sputtering

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Robust antimicrobial coatings are needed in many applications. However, standard eluting and organic materials suffer from environmental degradation. Mechanical rupturing of cell walls by nano-pillars has recently been hailed as an efficient bactericidal approach that works without external activation. This study compares the bactericidal efficacy of benchmark coatings and nano-patterned surfaces. A range of hard coatings based on TiN was found to possess a nano roughness of <25 nm (AFM) and a significant surface coverage by large scale macroparticles produced in a cathodic arc process. These coatings required light activation to achieve moderate antimicrobial kill rate of ~ 1 log for *Staphylococcus aureus* (*S. aureus*) and were inactive in the dark. TiN films prepared by High Power Impulse Magnetron Sputtering exhibited a strong plasmonic behaviour with low optical losses, as determined from the imaginary component of the electric permittivity obtained from ellipsometry, and a resonant wavelength in the infrared spectrum. Patterning by reactive ion etching and colloidal masking produced nano pillars with sub-micron diameters. Significant light activation of phonons and production of hot electrons was detected by Raman spectroscopy in patterned surfaces compared to the flat as-deposited films. The effect of these surfaces on bacterial growth in the dark and under visible light illumination has been studied by AFM and colony-forming unit (CFU) calculations. The mechanisms of killing will be discussed.

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# Imaging of individual HiPIMS pulses by high-frame-rate camera

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In high-power impulse magnetron sputtering (HiPIMS) plasma organizes into dense azimuthal regions, which form periodic or quasi-periodic patterns [1]. These structures known as spokes rotate with velocities of several km/s. They are not specific to HiPIMS discharges but also form in classical DCMS [2] and RFMS [3] discharges. In this work, we studied the plasma evolution of individual HiPIMS pulses using a high-frame-rate camera. A systematic investigation of plasma self-organization and spoke dynamics was performed for a range of argon pressures (0.25-2 Pa) and peak discharge currents (10-400 A). The experiments demonstrated that plasma evolves through three distinct stages as the discharge current increases [4]:

**Stage I**: From the current onset and up to ~25 A, spokes have an elongated arrowhead-like shape and propagate in the -**E**×**B** direction, the same as in DCMS discharges. The number of spokes is influenced by pressure and current growth rate. At the lowest pressure (0.25 Pa), most often a single spoke is observed, while at higher pressures (1-2 Pa) two-spoke patterns form. The spoke velocity depends on the current growth rate, number of spokes and pressure. Velocities range from 4-15 km/s for a single spoke and 1-9 km/s for two spokes.

**Stage II**: As current increases above 25 A, plasma starts to display chaotic behavior marked by aperiodic spoke patterns and irregular dynamics. Spokes often merge, split or form anew while they propagate either in retrograde or prograde direction.

**Stage III**: Above the current of ~50 A, ordered spoke patterns begin to emerge. In stage III spokes are azimuthally shorter, typically exhibit triangular shape, and rotate in the **E**×**B** direction. Overall, the spoke dynamics is less complex and is influenced primarily by pressure. At the pressure of 0.25 Pa spokes rotate with the velocity of 9 km/s, while at 2 Pa they rotate with the velocity of 6 km/s. Notably, the spoke velocity is largely unaffected by the discharge current and number of spokes.

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## Manufacturing of nanohole arrays for stable vaccine formulations

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Nanohole arrays have been investigated as a solution for vaccine enclosures without the need for cold storage. In this study, we employed electron-beam lithography (EBL) to create multiple hexagonally arranged nanohole arrays. We varied the wall thicknesses between each hole to achieve the maximum nanohole density.

Four patterns were designed and tested, with wall thicknesses of 100 nm, 40 nm, 20 nm, and 10 nm. Both positive resist (PMMA) and negative resist (ma-N) were used. The lowest achieved mean wall thickness after processing was 62 nm ± 16 nm, corresponding to approximately 5.7 × 10^8 holes per cm^2. This could enable high-capacity loading of vaccine-loaded lipid nanoparticles, potentially reducing the cost of vaccine storage and transportation, especially in developing countries. Such a development is crucial for improving global vaccination efforts and ensuring equitable access to vaccines worldwide.

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# Preparation of thin films by reactive multi-pulse magnetron sputtering

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The work deals with reactive deposition processes for the preparation of thin films of TiN and TiC using methods based on magnetron sputtering, namely direct current magnetron sputtering (dcMS), standard high-power impulse magnetron sputtering (s-HiPIMS) and multi-pulse high-power impulse magnetron sputtering (m-HiPIMS). It has been repeatedly shown that ionization degree of sputtered species and working gas is substantially higher in HiPIMS process in contrast to standard dcMS. Due to the highly concentrated power in time during the pulse the peak power delivered to the process is exceeding common dcMS power range. Therefore, the raised energies of particles in the vicinity of the target enable creation of the plasma with higher concentration of ionized species. The emphasis is in this work put on m-HiPIMS as an innovative and thoroughly studied technique aspiring to improve standard HiPIMS [3]. The m-HiPIMS method is based on arranging desired amount of high-power pulses in the pulse packages including pulses and short off-time intervals. Using such multi-pulse operation enables reaching even higher concentration of ionized particles due to pre-ionized plasma at the start of each of the subsequent pulses [2]. The coatings deposited by processes with high ionization tend to exhibit decreased roughness and high density [5], both being important features for wide range of industrial applications.

The experimental part of the work consisted of the deposition of coatings and the comparison of particular deposition settings in the case of the deposition process parameters, waveform investigation, surface roughness, crystalline structure and their mechanical properties. Analyzed data clearly demonstrate the differences among dcMS, s-HiPIMS and two m-HiPIMS configurations. Furthermore, the connection among the number of pulses, crystalline structure and hardness can be followed plainly. An extraordinary outcome of this work is the finding of obviously increasing hardness with the number of pulses in m-HiPIMS configuration. A decisive explanation for this behaviour is introduced in the theory of Hall-Petch effect and inverse Hall-Patch effect [1, 4].

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# Ion energy comparison between HiPIMS and bipolar HiPIMS for the generation of superconducting thin films

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Magnetron sputtering is a common technique used for the production of thin films in vacuum settings. The sputtered ions are ejected from the target material with high kinetic energy and deposited onto the substrate to form the coating. Thus resulting in an energy transfer between the bombarding particles and the substrate, affecting processes such as diffusion, film growth, or crystallization. By providing a better understanding of the plasma chemistry involved in this particular process, the energy of the ions formed could be tailored to optimise the production of superconducting thin films for SRF cavities.

In this study, the behaviour of the positive ions formed from a niobium (Nb) target was investigated through mass spectrometry, using both HiPIMS and bipolar HiPIMS. For this, the input parameters from the HiPIMS power supply unit were varied such as pulse width, duty cycle or power. Additionally, both reverse positive potential, positive pulse width, and positive delay were varied for the bipolar HiPIMS. Lastly, a Langmuir probe was employed to measure the IV-characteristics of the plasma formed.

We compared and discussed the main variation for both cases and highlight the optimal parameters to maximise the niobium ions energy for different pressure conditions, ranging from $1x10^{-3}$ mbar to $1x10^{-1}$ mbar.

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# The importance of materials surfaces in the fight against antimicrobial resistance

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Antimicrobial drugs have been one of medicine's greatest successes, evidenced by the countless lives they have saved. Moreover, such drugs are essential part of the surgical practice that greatly reduces the risk of infection in the majority of elective surgery, such as joint replacement, enabling massive improvement in quality of life for many people. Antimicrobial resistance (AMR) among pathogenic microorganisms has been known almost as long as antimicrobial drugs have been used. A combination of factors – including the diversity and evolution of bacteria, widespread and sometimes unregulated use of antibiotics, and the limited availability of new drugs – has led to an AMR crisis, where resistant pathogens threaten to become so widespread that antimicrobial drugs rarely be effective and most elective surgery will be too high-risk to contemplate.

Materials surfaces play a critical role in the problem – and in the solution. Solid surfaces harbour reservoirs of pathogens and hence are a substantial route for transmission of infections. For example, surfaces such as floors, door handles and bed rails within hospital wards harbour antimicrobial-resistant bacteria of the type that can cause infections of orthopaedic prostheses. The surface of the prostheses themselves provide an "unnatural" environment within the human body that allows bacteria to colonise and avoid destruction by the immune system. However, the same surfaces offer major opportunities to combat AMR. Coatings that release antimicrobial agents can help to minimise the amount of antimicrobials that have to be used and to allow use of antimicrobial molecules that are not suitable for systemic (e.g. oral or injected) use. Surfaces that kill pathogens physically or by light-activated chemistry offer a means of controlling them that is completely independent of antimicrobial drugs. Major interdisciplinary work – including coatings researchers and manufacturers, microbiologists, clinicians and surgeons – is needed to meet the Global Grand Challenge of AMR by providing novel antimicrobial solutions that will be effective in their own right and help to preserve the effectiveness of the antimicrobial drugs we have.

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# Solvent cleaning in high-tech-coating processes

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High-tech coating processes require much higher component cleanliness levels compared to conventional coating processes. Considering the wide range of contaminants that need to be removed, it is a major challenge to find the optimum cleaning process in terms of the cost-benefit ratio.

Here it is interesting to consider the previously underrepresented use of modern organic solvents, if necessary, in combination with aqueous cleaning media.

In this context, the important principle of cleaning technology must be taken into account: "like dissolves like".

This principle means that non-organic (polar) contaminants should preferably be cleaned with aqueous media and organic (non-polar) contaminants should preferably be cleaned with organic solvents such as modified alcohols or isoparaffins.

HEMO provides the market with tried and tested processes in combination with organic solvents from SAFECHEM.

SAFECHEM provides the solvents of the Dowclene and Dualene series, which are very well known in degreasing of parts. They are approved solvents in many industries, such as the aviation and automotive industries. These organic solvents are isoparaffins (hydrocarbons with chain lengths C9 to C14) or modified alcohols.

HEMO provides the process engineering systems. Depending on requirements, these are either pure solvent applications or, if necessary, hybrid technologies such as Beyond or Hybrid.

In the hybrid process, organic solvents and aqueous media with cleaning agents are used one after the other, but in the same working chamber. In the Beyond process, organic solvents are used in a mixture with demineralised water.

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# Focused magnetron sputtering: A high-density coating deposition for industrial applications

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Conventional coating techniques, such as Cathodic Arc Evaporation (CAE), encounter obstacles stemming from microscopic defects. In contrast, Focused Magnetic Field Magnetron Sputtering (F-MS) emerges as a transformative solution, achieving high plasma power densities for large-scale targets. F-MS outperforms conventional magnetron sputtering (DCMS) by achieving a six-fold increase in power densities [1]. This advantage, combined with efficient cooling and extended duty cycles, positions F-MS as a revolutionary technology seamlessly integrated into PLATIT's PVD coating unit Pi411, stands as a transformative advancement in the realm of hard protective coatings for industrial applications.

F-MS is realized by longitudinally moving a reduced-size magnetron inside a tubular target, enabling high-power sputtering even for large-scale targets [1]. In this contribution, the maximum peak sputter power density of 840 W/cm² was achieved using a DC power supply with a 25 kW output (the maximum utilized in this research) and a target with dimensions Ø110x510 mm [1]. While various target compositions exist, this research focuses on coatings produced using an alloyed AlCr target.

Coatings of (Al,Cr)N, deposited via F-MS, exhibit a stoichiometric composition, smooth surfaces, and controlled growth defect levels. Mechanical and cutting tests demonstrate hardness and wear rates comparable to or surpassing state-of-the-art CAE coatings [1]. Furthermore, cutting tests conducted through various methods such as trochoidal milling, climb milling, and gear hobbing confirm the robust performance of (Al,Cr)N coatings.

The introduction of F-MS technology signifies a transformative leap in the coating industry, addressing challenges posed by traditional methods. The ability to achieve high plasma power densities, even for large-scale targets, positions F-MS as a game-changer, offering coatings that not only match but exceed the performance of current state-of-the-art CAE coatings [1]. This technology holds immense potential for advancing coating industry practices, introducing heightened efficiency, and driving innovation in applications such as microtools, gear hobbing tools, and shank tools.

**Keywords:** Movable magnetron, AlCrN, Industrial-Scale Magnetron Sputtering, Plasma Power Densities.

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## Poster 1

# A novel high-efficiency plasma nitriding process utilising a HIPIMS discharge

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Lifetime and biocompatibility of orthopaedic implants are crucial in meeting the new challenges brought about by the fall in patient age and the ageing population. The high-load surfaces in contact with the biological environment must display enhanced tribological properties, biocompatibility and reduced metal ion release in long-term clinical performance. Surface modification techniques such as nitriding, can significantly improve the in-service behaviour of the medical grade alloys in current use. We report on a novel approach for nitriding of CoCrMo alloys using High Power Impulse Magnetron Sputtering (HIPIMS) discharge. The new nitriding process has been successfully carried out at the National HIPIMS Technology Centre at Sheffield Hallam University, UK in an industrial size Hauzer 1000-4 system enabled with HIPIMS technology. While the nitriding ion flux is controlled by the HIPIMS magnetron plasma source, the ion energy can be independently set via the substrate bias. Implementing the HIPIMS source allows reducing the operational pressure by one order of magnitude compared to conventional dc plasma nitriding (DCPN). Energy-resolved mass spectroscopic plasma analyses (Fig. 1) have identified significantly enhanced production of ions of molecular nitrogen ($N_2^+$), atomic Nitrogen ($N^+$) and $N_2H^+$ radicals in the HIPIMS discharge compared to DCPN. Because of the low pressure of operation of the HIPIMS process, the energy of ions is similar to the bias voltage. In contrast, severe losses in ion energy is observed during DCPN due to the high operating which cause scattering collisions within the sheath. The high flux and high ion energy are primarily responsible for achieving a fourfold increase in process productivity as compared to state-of-the-art plasma nitriding processes (Fig. 2). The nitrided surface layers exhibit excellent mechanical and tribological properties, which bring about significant improvements in hardness, fracture toughness and wear. The protective function of the nitrided layer against corrosion in the aggressive environments of simulated body fluid are remarkably augmented. The barrier properties of the nitrided layer have been demonstrated through a reduction in metal ion release by as much as a factor of 2, 4 and 10 for Co, Cr and Mo respectively.

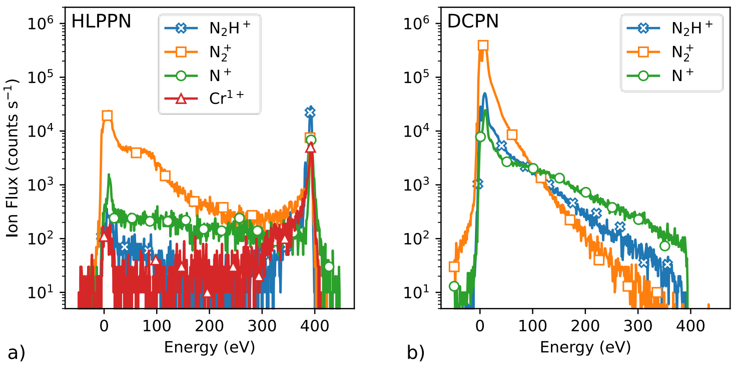


Fig. 1 Ion energy at the surface of nitrided components in a)HLPPN and b) DCPN

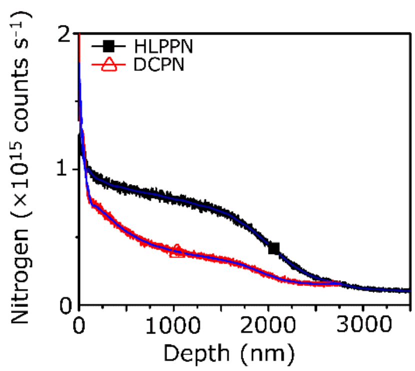


Fig. 2 Nitiriding case depth for HLPPN and DCPN

**Keywords:** HIPIMS, Low-Pressure Plasma Nitriding, Duplex treatment, Hardness, Corrosion

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## *Poster 2*

# Mechanical and anticorrosive properties of (TiAlZrTaNb)Nx high entropy nitride thin films deposited by HiPIMS

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We studied (TiAlZrTaNb)Nx coatings deposited on superalloys and Ti alloy substrates by means of the high-power impulse sputtering (HiPIMS) technique on substrates. The coatings were deposited under identical deposition conditions, that is, the same substrate temperature, discharge power, pressure, substrate-to-target distance, and Ar/N 2 ratio flow-rate. The films' microstructure and chemical composition were analyzed via X-ray diffraction (XRD) and energy-dispersive X-ray spectroscopy (EDS) probe. The corrosion resistance was studied via the polarization potentiodynamic polarization method employing a 3.5% by weight NaCl solution and the mechanical properties were evaluated by means of nanoindentation. The mechanical and corrosive behavior of the films deposited were compared depending on the type of substrate used. Also, the relationship between the growth conditions, microstructure, electrochemical and mechanical properties is presented and discussed.

**Keywords:** HiPIMS, TiAlZrTaNbNx, Hardness, Corrosion

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## Poster 3

# Magnetron sputtered β-$Ti_3AuAg$ coatings for long lasting artificial joints and beyond

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Artificial joint replacement surgeries have drastically improved the quality of life for millions of patients suffering joint failure caused directly or indirectly by accidents, increasing life expectancy, and/or degenerative medical conditions. However, a significant number of these implants fail post replacement procedure and require revision surgery. This painful and invasive procedure comes as a significant burden on public health expenditure. Within the UK itself, the cost of revision surgery is expected to rise from £75 M/year to £200 M/year between 2020- 2040. Some of the primary reasons for the failure of these implants are aseptic loosening, post-surgery infections, and adverse reaction to leached toxic elements. Articulating surfaces of joint implants are commonly made from medical grade Ti6Al4V (or Ti64) alloy, which has excellent mechanical properties and biocompatibility but can suffer from poor wear performance leading the release of harmful metallic ions like Al and V. The current lifetime of orthopaedic implants can be extended by coating these surfaces with thin films of harder biocompatible alloy systems, thereby reducing the need for costly reconstruction surgery. This work has developed a multi-functional biomaterial with enhanced hardness, superior wear resistance, and antimicrobial functionality to combat these various failure mechanisms and increase the life cycle of these implants within the human body.

In this work, thin films (~1µm) of Ti(1-x) Au(x)Ag alloy were deposited on Ti64 substrates by the magnetron sputtering technique. The elemental composition, stoichiometry, and deposition conditions were optimised to develop a Ti-Au-Ag intermetallic with enhanced mechanical properties that were performance tuned by suitable thermal activation processes like in-situ substrate heating and by controlling the energy of the adatom species through control over deposition pressure. The first phase of this work achieved a surface hardness value of 8.8 GPa on the $Ti_3Au$ intermetallic coating, which is 200% higher than the bare Ti64 substrates (~4 Gpa). With further material processing and fine tuning of deposition parameters this value was enhanced to 11.9 GPa. Ag doping further increased the mechanical hardness of this intermetallic to over 14 GPa, as doping sites act as defects inhibiting the movement of dislocation planes through the $Ti_3Au$ matrix. The improvements in mechanical performance were corelated to corresponding changes in the structural (XRD), morphological (SEM) and chemical (EDX/SIMS) nature of the Ti-Au thin films, supporting the emergence of super-hardness through the development of the β phase of the $Ti_3Au$ intermetallic. The safe (non-cytotoxic) profile of these $Ti_3AuAg$ thin film coatings was confirmed with >90% viability of Mouse fibroblast cells (L929), whereas the extremely low levels of leached ion concentrations (<0.1 ppm) confirm their biocompatible nature. This foundational study was successful in developing Ag doped $Ti_3Au$ intermetallic thin films with mechanical hardness values greater than 14 GPa, a 200% improvement in wear resistance compared to uncoated Ti64 bare substrates and antibacterial resistance against the strains of *E. Coli* bacteria. The potential of such a multi-functional super hard biocompatible coating extends beyond just joint implants, to applications ranging from hard wearing smart jewellery to human exoskeleton for extreme environments.

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## Poster 4

# Assessment of slurry-erosion and aqueous corrosion behaviour of Niobium as an alternative cladding material for neutron producing targets

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The premature failure of tantalum-cladded tungsten sources used for generating neutrons at the ISIS Neutron and Muon Source at the Rutherford Appleton Laboratory (RAL) has compelled scientists to lookout for alternative cladding materials. The failure is attributed to erosion-corrosion resulting from the circulating cooling water. In this study, the slurry erosion response of Nb as a potential candidate material was analysed using a home-built impinging jet erosion-corrosion apparatus. X-ray fluorescence spectroscopy and x-ray diffraction studies respectively confirmed its purity as 99.8% and a body-centred cubic structure with (110) prominent peak orientation. Pure slurry erosion studies at different slurry concentrations of 3, 5, 7, and 9 wt.% at an impingement angle of 30° and a velocity of 6 $ms^{-1}$ determined the peak erosion rate to occur at 7 wt.% concentration. The studies were extended to find the effect of impact angle on erosion rate by conducting experiments at different impingement angles (20°, 30°, 45°, 60°, and 90°), a constant slurry concentration of 7 wt.% and velocity of 4 $ms^{-1}$. The results revealed the highest erosion rate at an impact angle of 30°, which is consistent with results typically observed for most ductile metals. Erosion mechanisms were determined by analysing scanning electron microscopy images of the erosion scars at impact angles of 30° and 90°. At an impact angle of 30°, the erosion mechanism consisted of a dominant micro-cutting action of the plastically deformed material resulting from a continuous influx of erodent particles having irregular shape and sharp edges. Whereas plastically deformed material in the form of lips and its breakage due to fatigue caused from repeated impacts constituted as the main erosion mechanism at 90° of particle impact. Static aqueous corrosion studies were also performed under potentiodynamic conditions at different pH values; pH 3 and pH 5 (0.5M citric buffer solutions) and pH 7 (3.5% NaCl solution) using a three-electrode cell system. Nb samples exhibited a strong tendency to passivate at all pH conditions. A more noble $E_{Corr}$ value was observed for pH 3 compared to NaCl, however citric buffer solutions resulted in higher corrosion current densities ($2.91\times10^{-4}$, $1.23\times10^{-4}$, and $8.47\times10^{-5}$ $mAcm^{-2}$ for pH 3, 5, and 7, respectively). The results from this study are novel as there is no literature on the slurry erosion of Nb and will act as significant feedback for the design of future target materials at RAL.

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## Poster 5

# Influence of pulse duration on plasma chemistry and thin film growth of plasmonic Titanium Nitride deposited by constant current regulated HIPIMS.

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This study documents the results of an investigation into the effect of pulse duration within HIPIMS discharges, specifically investigating the effects on plasma chemistry, temporal evolution and on the changes to thin film texture of films produced from these discharges. Pulse durations ranging from 40-200µs were studied. Time-Averaged Optical Emission Spectroscopy has been conducted on a series of discharges with different pulse durations and multiple species identified within these discharges have been studied using Time-resolved Optical Emission Spectroscopy to study how they develop throughout the pulse. Emissions of unreactive gas, reactive gas and metal species within the discharge have been measured and studied. The data obtained from the Time-Resolved Optical Emission Spectroscopy shows three stages that can be used to characterise the generation of the discharge: known as Gas Rarefaction, Pumping and Steady State. The studies also show a development from gas dominated to metal dominated, there is also shown to be an increase in electron temperature within the discharge even when current and voltage remain constant. Titanium Nitride films have also been produced at room temperature from the different discharges studied to investigate the role that pulse duration and plasma chemistry plays on the texture of the produced films. These films have been shown to be plasmonically active when exposed to electromagnetic radiation within the visible spectrum.

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## Poster 6

# Structural, morphological and topographic characterization of (TiAlZrTaNb)Nx coatings deposited by HiPIMS

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In this study, TiAlZrTaNb nitride thin films were deposited on silicon and metals substrates by High-Power Impulse Magnetron Sputtering (HiPIMS) modifying polarization and pressure to change the energy of the ions. The effect of substrate bias and working pressure on structure, morphology and structure of the films was investigated. The microstructure, morphology and chemical composition of the coatings were analyzed by X-ray diffraction, scanning electron microscopy, atomic force microscopy and energy dispersive X-ray spectroscopy. The effect of the deposition parameters is highlighted in order to further enhance HiPIMS coatings properties. Finally, the growth mechanism in the TiAlZrTaNbNx films will be discussed in this work.

**Keywords:** HiPIMS, TiAlZrTaNbNx, microstructure, bias, pressure

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## *Poster 7*

# Fabrication of high temperature Kinetic Inductance Detectors (KID's) using High Power Impulse Magnetron Sputtering.

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In astronomy, Superconducting Kinetic Inductance Detectors (KID's) have emerged as a popular technology for detecting light in the millimetre to submillimetre wavelength range [1]. Their main advantage over existing superconducting technologies (e.g. Bolometers) is that they can easily be multiplexed, allowing large arrays in excess of 10,000 pixels to be read out with relatively little complexity [2]. However, typical superconductors such as Aluminium require cooling to sub-K temperatures, dramatically increasing the cost and complexity of a detector system.

Recent research into KID's has focused on fabricating them out of higher temperature superconductors such as Titanium Nitride (TiN) [3–5] which has a controllable transition temperature varying from 0.05K - 5K based on the film stoichiometry [6]. This controllable range enables the detector to be tuned for a given application (wavelength and/or background loading) with the potential to reduce cryogenic complexity for high background applications. However, getting reproduceable films on large wafers has been shown to be challenging, with sputtered films being very sensitive to changes in both stoichiometry and thickness[6], making large scale arrays very difficult to fabricate.

In this work we demonstrate how superconducting films of TiN and Nb with exceptional uniformity can be produced using a 8" compatible, custom designed sputter system with a HIPIMS power supply. We compare films made using HiPIMs as well as DC and show how their superconducting properties can easily be tuned. Finally, we present and analyse RF measurements of KID arrays fabricated using this technique.

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