



innovaTiAl

**"Innovative processes and materials to synthesise
knowledge-based ultra performance nanostructured
PVD thin films on gamma titanium aluminides"**

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Reactive Sputtering, Growing Rapidly in the Industry

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Reactive sputtering has been used in e.g. tool coating applications, but has lost position to other technologies like Cathodic Arc in the early nineties. Parallel a new market application has developed; coatings on components. The main driver has been the wider application of Diesel engines, in combination with more stringent emission regulations and better fuel efficiencies. ARC coatings have the disadvantage of an increased surface roughness (due to incorporation of droplets). Purely sputtered coating systems like a-C:H:W (or W-DLC) and Cr₂N have been applied on a wide scale.

Pure PACVD systems like a-C:H:Si (Si doped DLC) or a-C:H (DLC) have been applied as well. Presently the trend is clearly to hybrid systems, whereby reactive sputtering is used to build a supporting layer, followed by a top coating grown by PACVD.

The technologies are not out-developed yet, further progress can be expected in two directions:

- The deposition technologies used are aiming for an enhancement of ionization, one of the promising technologies that will be discussed is High Power Pulsed Magnetron deposition.
- The composition of the coatings is further developed, especially the multi-layer structure will be developed further. Nanolayered coatings have demonstrated to have better mechanical properties.

The Use of PVD Coatings in Medical Applications

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The rapid progress of medical technology and procedures requires a constant search for both the improvement of the current state of the art and 'leaps' forward in related developments.

Material applications, new material usage, design and advanced surgical procedures demand even more from existing medical technologies. It is here where PVD coatings can supply both enhancements to existing applications and be an integral part of new developments and designs. Ionbond's medical coatings division, Medthin, coats a wide range of instruments, prostheses and implant tools for both the medical and dental markets.

The choice of a particular coating depends on the requirements of the application and the surface properties needed, which can range from:

- wear resistance
- metal ion release reduction
- anti-reflectivity
- low coefficients of friction
- corrosion resistance
- colour contrast
- promotion of tissue growth to the implant
- anti-allergic

The PVD coating must be biocompatible and be reproduced to the highest quality standards as demanded by the medical industry. Consideration must also be taken not only of the environment of use, but also complementary factors, such as the resistance to repeated high pH wash cycles, radiation, high sterilisation temperatures and UV light resistance.

The scope for the use of PVD coatings ensures that careful consideration must be taken with regards to the correct coating material (metal, alloy, pseudo-ceramic), the coating design (mono-, multi- or nano-layer), the substrate and the method of deposition.

The above, plus the use of other complementary technologies to enhance the performance of deposited thin film technologies, will be discussed.

Pathway of Texture Evolution in Nitride Multilayer Coatings Deposited on Polycrystalline Stainless Steel Substrates

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Multilayer coatings of CrN and Y doped AlCrN layers have been deposited by the combined HIPIMS/UBM technique on austenitic stainless steel (Cr1.19Fe0.7Ni0.11) substrates covered by AlCrN underlayer at 450°C substrate temperature. The substrates were prepared by mechanical polishing. Prior to the deposition of the underlayer the substrate surface was treated by HIPIMS Cr⁺ bombardment, [1, 2]. During deposition -75V, -90V, -120V or -150V bias voltages were applied. The structure of the films was investigated by X-ray diffraction methods (Bragg-Brentano and pole figure analysis) and cross sectional transmission electron microscopy.

The AlCrN underlayers have generally local orientational relationship to the individual crystals of the polycrystalline substrate. The detailed analysis of the interface regions between selected substrate crystals and the locally grown underlayer indicated that epitaxial or topotaxial relations can exist between the substrate crystal and the underlayer in the most cases. In case of epitaxy the underlayer is single crystalline, while in case of axiotaxy it is polycrystalline with columnar structure. The growth of these oriented AlCrN underlayer crystals, depending also on their orientation, is usually interrupted, new crystals nucleate and start a competitive growth. This sets in either already within the underlayer or at the underlayer/multilayer-coating interface. This results in a transition thickness range with more or less random orientation. The competitive growth of these randomly oriented crystals is determined by the deposition conditions and results in the formation of V-shaped crystalline columns of the multilayer structure with [220] or [111] texture.

[1] W.-D. Münz, A.P. Ehasarian, P.Eh. Hovsepian, EP 02 011 204.1 (2001).

[2] A.P. Ehasarian, P.Eh. Hovsepian, W.-D. Münz, US 10718435, (2005).

Transmission Electron Microscopy and Electron Energy Loss Spectroscopy Study of C/Cr Coatings

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Carbon based coatings have attracted great interest in a wide range of industries due to their excellent properties such as low friction coefficient and good wear resistance. Multilayer C/Cr coatings have been produced by unbalanced magnetron sputtering. Resultant specific wear rates of $10^{-17} \text{ m}^3\text{N}^{-1} \text{ m}^{-1}$ were recorded with a friction coefficient of 0.17. The structure of the C/Cr coatings, and therefore the tribological properties, is strongly influenced by the deposition bias voltage. The coatings evolved from an amorphous (-65V to -95V) through an 'onion-like' structure (-120V) to nanoscale multilayer structure through a self-organising mechanism, not related to substrate rotation (-350V and -450V), and finally a uniform fine grain structure (-550V). The changes in structure were associated with friction coefficient variations from 0.22 (-65V) through 0.16(-95V) to 0.31 (-350V). The structure evolution is believed to be a result of ion bombardment and elemental diffusion.

In order to further understand the ion irradiation induced self-organising mechanism, the multilayer C/Cr structure at -350V and amorphous structure at -75V were examined using a dedicated spherical aberration corrected scanning transmission electron microscope (STEM), coupled with electron energy loss spectroscopy (EELS), which is capable of sub angstrom (\AA) high angle annular dark field (HAADF) resolution. The multilayer was composed of alternating C-rich layer (C-rich clusters) and uniform Cr-rich layer. Line scans across the multilayers and along the C-rich clusters were performed using a 0.1nm probe. Quantitative EELS analysis and profiles showed the variation of C and Cr. High resolution EELS near edge fine structure indicated that the C bonding was amorphous carbon, consistent with the high resolution electron micrograph. In addition, the ratio of Sp^2 (π bond) and Sp^3 (σ bond) across the layers and along the clusters are presented and discussed. Amorphous structure coating was composed of columnar grains. The grain boundaries were C-rich, and grains amorphous.

Sputter Deposition of Cubic Boron Nitride Tool Coatings

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In contrast to diamond, cubic boron nitride (cBN) does not react with iron at elevated temperatures. Therefore cBN is highly promising for cutting tool applications, especially for machining of iron containing materials like steel. Although bulk cBN, prepared by high pressure – high temperature synthesis, is well established in industrial practice, it seemed to be impossible until about 10 years ago to prepare sufficiently thick ($> 1 \mu\text{m}$) cBN coatings. Due to extremely high compressive stresses and additionally a pronounced sensitivity against humidity, most cBN films delaminated already with thickness of 0.1 to 0.3 μm . However, in the last years some few research groups in the world were successful in deposition of "thick" cBN coatings ($> 2 \mu\text{m}$), but more or less only on silicon wafer substrates. In our laboratory such thick cBN coatings were deposited by reactive sputtering using a boron carbide target. Following a boron carbide interlayer, a gradient layer with continuously increasing nitrogen content was prepared. After the cBN nucleation the process parameters were modified for the cBN film growth. The transfer of this deposition process from silicon to technically relevant substrates like cemented carbide cutting inserts required some essential process modifications: (i) thicker boron carbide layers and (ii) additional intermediate layers, consisting of metal (Ti) or metal nitrides (TiN, TiAlN). Such design conceptions allowed to prepare several μm thick, long time stable cBN based coatings on cutting inserts. Oxidation experiments revealed a stability of the coating system on cemented carbide up to 700 °C and higher. Coated cutting inserts were tested in turning and milling operations for different metallic workpiece materials. The test results will be compared to those of well established cutting materials and tool coatings. The deficits of the actually available coating systems and deposition techniques as well as requirements to future developments will be discussed.

VMeCN Based Superlattice Coatings Deposited by the Combined HIPIMS/UBM Technology for Machining of Al and Ti-alloys

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New VMeCN based PVD coatings, [1] combining high hardness, low friction coefficient and chemical inertness have been developed for machining of "sticky" (Al, Ti and Ni based) alloy materials used in Aerospace, Automotive and Medical Industries.

Excellent performance was achieved due to the synergy between V and C as main coating elements and the nanoscale multilayer structure of the coating. Vanadium provides for low coefficient of friction due to the formation of low melting point, highly lubricious V_2O_5 during sliding wear. The role of Carbon is to further stabilise the friction coefficient, ($\mu = 0.37$ against Al_2O_3) to increase the chemical inertness between cutting tool and workpiece material and introduce a specific wear mechanism.

The high hardness ($H_V = 2700 \text{ kg/mm}^2$) and unique wear mechanism, result from the nanoscale multilayer/superlattice structure, (bi layer thickness $\Delta = 2.2 \text{ nm}$) of the coating. TiAlCN/VCN has been deposited by the combined High Power Impulse Magnetron Sputtering / Unbalanced Magnetron Sputtering, (HIPIMS/UBM) technology using a Hauzer HTC 1000-4 PVD system. Macroparticle free V^+ ion flux has been generated by HIPIMS discharge to sputter clean the substrates prior to the coating deposition, [2, 3]. TiAlCN/VCN has been deposited by unbalanced magnetron sputtering using TiAl and V targets in a mixed CH_4 , N_2 and Ar atmosphere.

Coatings phase and nanoscale structure were characterised using a variety of surface analysis techniques, such as low and high angle X-ray diffraction analyses, cross-section transmission electron microscopy and EELS. Atomic resolution STEM showed that the HIPIMS surface pre-treatment produces extremely clean coating-substrate interface and promotes local epitaxial growth. EDX line scanning carried out in a STEM across the interface revealed that V has been implanted in the substrate to a depth of several nanometers during the HIPIMS etching process, which provides for the excellent adhesion.

STEM bright field and Z-contrast imaging combined with EELS analysis revealed that Carbon was segregated during the coating growth at the columnar boundaries but more importantly laterally at the interfaces between the individual layers of the superlattice structure. The lateral segregation of Carbon produces lower shear strength interfaces, which is a unique feature of the TiAlCN/VCN superlattice. It has been shown that these types of interfaces provide for controlled on a nanometer scale wear mechanism and prevent formation of thick built up layers in machining of „sticky“ alloys. The incorporated Carbon in the V based superlattice plays a dual role. In case of machining of Al based alloys, Carbon inhibits metallurgical reactions with the workpiece material, whereas with Ti based alloys, the low shear strength interface prevents formation of thick built up layers.

Cutting tests demonstrated the excellent wear behaviour of the VMeCN based superlattice coatings. In dry milling of Al 7010-T 7651 alloy, TiAlCN/VCN outperformed state of the art Diamond Like Carbon (DLC, Cr/WC/a-CH) coatings by factor of 4. In turning operations,



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cemented carbide inserts coated with TiAlCN/VCN produced 2-3 times more Ti-alloy acetabular cups (orthopaedic implants) compared to uncoated tools.

[1] *P. Eh. Hovsepian, A. P. Ehasarian, T. Deeming. PVD Coated Substrate, Pat. Appl. B0508485.0 filed 27 April 2005 .*

[2] *W.-D. Münz, A.P. Ehasarian, P.Eh. Hovsepian, EP 02 011 204.1 (2001).*

[3] *A.P. Ehasarian, P.Eh. Hovsepian, W.-D. Münz, US 10718435, (2005).*

Tribofilm Formation and Wear Mechanisms of TiAlCN/VCN and TiAlN/VN Coatings Characterized by Analytical TEM

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Dry sliding wear tests using a counterpart of alumina ball were carried out on TiAlCN/VCN [1] and TiAlN/VN nanoscale multilayer/ superlattice coatings respectively. In order to study the wear mechanisms taking place in the area of the sliding contact, analytical transmission electron microscopy imaging, energy dispersive X-ray spectroscopy and electron energy loss spectroscopy were employed to investigate the worn surfaces. The TEM analysis revealed the formation of a 20 – 50 nm thick, well adhered to the worn surface of TiAlN/VN tribofilm. Combined EDX and EELS analyses indicated that the tribofilm was predominantly an amorphous mixture of multicomponent oxide although some remaining nitrogen was also detected. In particular, the fine near-edge structure in the N-K edge suggested the existence of nitrogen-depleted nitride in the tribofilm, which indicated tribo-oxidation taking place through decomposition of nitride wear debris. Interestingly, the tribofilm seemed to provide good protection of the TiAlN/VN from mechanical wear such as deformation, cracking and delamination. On the worn surface of the carbon-containing TiAlCN/VCN coating, however, such well-adhered tribofilm was not found, revealing reduced adhesive bonding between the coating and the wear debris. The TiAlCN/VCN coating was therefore exposed to a tribofilm- free sliding contact to the counterpart, which resulted in the generation of nm-scale delamination fragments and excellent overall wear resistance. EDX and EELS analyses of the wear debris indicated that tribo-oxidation was the predominant wear mechanism.

[1] P. Eh. Hovsepian, A. P. Ehasarian, T. Deeming. *PVD Coated Substrate*, Pat. Appl. B0508485.0 filed 27 April 2005 .



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Wear Resistant Hard Coatings of Using Closed Field Unbalanced Magnetron Sputtering Technology

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A deposition process of using closed field unbalanced magnetron sputter ion plating technology has been fully developed to produce wear resistant hard coatings. This process uses multiple magnetron sources and elemental metal target with flexibility of depositing coatings of single metal element as well as multiple metals with nano-scale multilayers. The deposition process starts with an argon plasma ion clean using low power on the magnetron sources and high bias voltage on the substrate. And then a metal adhesive layer is deposited using high power on the magnetron sources and low bias voltage on the substrate. Afterwards the reactive nitrogen gas is introduced to produce a metal nitride and then additional metals are introduced to deposit a gradient multi-component nitride hard coating leading to a consistent nano-scale multilayer nitride at the top of the coating. Rockwell indentation, scratch, pin-on-disc, post heat treatment, and accelerated drilling testing are used to evaluate the coating performances. All the testing results indicate that the coatings are characterized to have excellent adhesion, high hardness, high wear resistance and high thermal stability and these properties can be explained as the reasons that coated drills have exceptional cutting performance.



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Mapping Erosion-Corrosion Mechanisms of Superlattice CrN/NbN Coatings

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There has been increasing interest in recent years in assessing the combined effects of erosion and corrosion on PVD coatings. In such cases, the interaction may be very complex. Erosion may enhance the corrosion rate of the coating by removal of surface corrosion products. In addition, corrosion of the coating, if it results in the formation of an adherent film, may provide resistance to wear on the coating surface.

In this work, the erosion-corrosion resistance of a CrN/NbN superlattice coating was evaluated at a range of applied potentials in an aqueous slurry environment. The effects of velocity and impact angle were investigated in such conditions. Scanning Electron and Atomic Force Microscopy were used to identify the mechanisms of damage on the surface.

The results indicated that the wear-corrosion mechanisms changed significantly as a function of the impact and corrosion conditions. Mechanistic changes and extent of synergism and antagonism of the erosion-corrosion interaction were identified on erosion-corrosion maps. Such trends in performance together with the potential of such maps to optimise coatings for erosion-corrosion performance are addressed in this presentation

Industrial Production PVD coatings for Tools and Components

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Since the initial laboratory based development of PVD coatings, the industry has developed a day-to-day production tool, implemented in a supply chain with an added value of more than one billion US\$/year.

Besides the initial applications for tools and thereafter for decorative applications, coatings are used more and more for automotive components in heavy duty applications.

Whereas in tooling a variety of different pieces of high individual value each are to be coated, it will be shown that the most favourable solution in that case will be a moderate size coating unit, whereas in decorative and component industry millions of parts of smaller size and less value per part will yield an optimum of big machines with solid repeatable processes.

Additionally the entire production flow must carefully be considered to achieve high quality and with that a reliable cost effective mass production.

The experiences and conclusions of Hauzer as a provider of high quality PVD-equipment and processes will be discussed in this talk.

Influence of the Negative Oxygen Ions on the Structure Evolution of Transition Metal Oxide Thin Films

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The energy distributions of O^- ions of magnetron sputtered Nb, Ta, Zr and Hf in an Ar / O_2 atmosphere were measured as a function of the oxygen partial pressure. Three ion populations were detected in the plasma: low, medium and high energy ions. The ion energy distribution functions were compared to distributions obtained based on sputtering theory. If the surface binding energy is assumed to be equal to the heat of formation, good agreement between the experiment and theory was achieved. From correlating the measured ion energy distributions with previously published phase stability data [Ngaruiya et al., Appl. Phys. Lett. 85(5), 748 (2004)], it can be deduced that large fluxes of medium and high energy O^- ions enable formation of crystalline transition metal oxide thin films during low temperature growth. The here presented data may be of general relevance for understanding the structure evolution of thin oxide films.

Oxidation, Embrittlement and Fatigue of Coated Titanium Alloy Ti-6-2-4-2S

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Due to their high affinity to oxygen, titanium alloys have poor oxidation resistance and tend to embrittlement when exposed to oxidizing atmospheres. Protection of titanium alloys by coatings is a promising way to improve environmental resistance, however, this is often achieved at the expense of mechanical properties, particularly fatigue resistance. The fatigue debit is often higher than can be accepted for technical applications and this has hindered the use of coatings for this purpose as yet.

Titanium alloy Ti-6-2-4-2S, an alloy currently used in aero engine applications at moderately elevated temperatures, was coated with two different types of metallic coatings from the Ti-Al-Cr and the Ti-Al-Ag system by magnetron sputtering and by a ceramic layer using an electrochemical coating process. Coated samples and uncoated reference samples were isothermally exposed to air at 600°C for 10, 100 and 1000h, respectively. Subsequently, room temperature fatigue tests were conducted using rotating beam loading at R=-1, f=100Hz. Cross-section micro-hardness measurements after testing as well as careful metallographic investigations using SEM revealed significant differences of the coatings in terms of oxidation behaviour and embrittlement as well as fatigue behaviour. The relevant mechanisms causing fatigue failure will be highlighted. Clearly, for practical applications, a fatigue debit due to the presence of coatings must be considered which presently appears to be 10% at best.

Pulsed Reactive Magnetron Sputtering of ITO Thin Films: Cross-Magnetron Effect and Film Property Distribution.

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Transparent conductive indium tin oxide (ITO) films for low cost applications were deposited by a reactive dual magnetron sputter process using metallic targets. The magnetrons were equipped with rectangular ($130 \times 400 \text{ mm}^2$) In:Sn targets (90 wt.-% In / 10 wt.-% Sn). A sine wave power supply was used at a frequency of about 70 kHz. All experiments were done in the transition mode in an argon-oxygen mixture.

The films were deposited onto silicon and float glass substrates. With moving substrates (dynamic deposition mode) we received good quality films with a deposition rate of about 100 nm \times m/min at an average power of 2 kW per cathode. The films had an electrical resistivity of $1.2 \times 10^{-3} \text{ }\Omega\text{cm}$, a refractive index of about 2.05 and an optical absorption of $k = 10^{-2}$ and thus were suitable for most low-cost applications.

When the substrates were fixed in front of the targets during deposition (static mode) films were inhomogeneous in both structure and properties. At some points the films had much better values (e.g. a minimum resistivity as low as $6 \times 10^{-4} \text{ }\Omega\text{cm}$ without intentional heating) showing the actual potentials of the process.

We have investigated the inhomogeneity of the deposition by laterally resolved measurement of both process parameters and film properties. From these results, the dependence of film structure and properties on certain process parameters was established and conclusions drawn how to overcome the inhomogeneity of the deposition and, hence, improve the overall film quality during dynamic deposition.

High Temperature Response of Nitride Coatings to Oxygen/Sulphur Containing Environments

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There are many situations where high temperature sulphidation is a serious problem. The wide-spread occurrence of sulphur in some fossil fuels and the presence of sulphur oxides and other sulphur-containing species in many combustion atmospheres derived from fossil fuels promote unavoidable damage due to sulphidation. Sulphidation attack is particularly severe in environments of low oxygen ($p_{O_2} \sim 10^{-18}$ Pa) and high sulphur ($p_{S_2} \sim 10^{-1}$ to 10^{-3} Pa) potentials at temperatures $>700^\circ\text{C}$. Such a mode of attack cannot be substantially inhibited even by the highly developed oxidation resistant alloys and coatings.

In environments of low oxygen and high sulphur activities, sulphides formed on certain alloys may offer a moderate measure of protection, in fact analogous to a protective oxide film, but in general, sulphide scales are much more friable and more subject to exfoliation than oxide scales on most high temperature alloys due to larger Pilling-Bedworth ratios. Furthermore, oxide scales formed on the alloys usually melt at relatively high temperatures, above the melting point of the alloys, whereas sulphides have comparatively low melting points and frequently form low melting point eutectics. In addition, the diffusion coefficients of cations in sulphide scales are relatively high because of the greater degree of non-stoichiometry of sulphide structures.

Enhanced sulphidation resistance may be achieved by employing the same principles used in oxidation resistant materials. The incorporation of appropriate elements into the base materials undergoing selective sulphidation leads to the development of a barrier layer capable of sustaining lower ionic transport rates. On the other hand, refractory and reactive metal nitrides generally possess high melting points and low dissociation partial pressures. These properties indicate that the refractory and reactive metal nitrides are highly stable compounds and can be used in coating forms to provide barriers against diffusion and high temperature environmental degradation.

This paper discusses the sulphidation/oxidation performance of several selected nitride coatings, single and multilayer, deposited on Ti and TiAl-based alloys at $750\text{-}850^\circ\text{C}$ in environments of low oxygen and high sulphur potentials.

Spinodal Decomposition of $Ti_{1-x}Al_xN$: Experiments and Modelling

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We have recently reported, that metastable NaCl-structured (c) $Ti_{1-x}Al_xN$ exhibits spinodal decomposition during annealing before phase transformation into the stable constituents c-TiN and wurtzite-structured (w) AlN. Here, we present results for the details of the decomposition process using a combination of experimental (small-angle neutron scattering (SANS), TEM, XRD, DSC, and nanoindentation) and theoretical (*ab initio*, continuum mechanics) methods.

During annealing of c- $Ti_{1-x}Al_xN$ at 900 °C for 1.5 h coherent \sim 3-nm-sized c-AlN and c-TiN precipitates are formed initiated by fully coherent Al-rich and Ti-rich domains. The intermediate metastable phases act as additional obstacles for dislocation movement, which is consistent with the hardness increase observed during annealing. As soon as the equilibrium phases are obtained and the precipitates coarsen, the film hardness decreases. Thus, age hardening and tempering is demonstrated for a ceramic thin film system. The calculated temperature-composition phase diagram shows a huge spinodal miscibility gap. At \sim 900 °C the difference in energy of formation for the decomposition of c- $Ti_{0.34}Al_{0.66}N$ into c-AlN and c-TiN is \sim 26.18 kJ/mol. Continuum mechanical investigations show that the average value of stored elastic strain energy for the decomposition is \sim 2.42 kJ/mol (as c-AlN and c-TiN have different specific volumes and elastic constants). The coherent surface energy contribution related to 3-nm-radius precipitates is \sim 1.95 kJ/mol. Thus, the overall enthalpy change during decomposition is reduced to \sim 21.81 kJ/cm³. The theoretical results are in agreement to the corresponding exothermic reactions of \sim 20.49 kJ/mol, as detected by DSC.

Influence of Y-addition on Microstructure and Mechanical Properties of $Ti_{1-x}Al_xN$ Hard Coatings

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$Ti_{(1-x-y)}Al_xY_yN$ coatings were deposited by reactive unbalanced magnetron sputtering on Si(100) from powder metallurgically prepared $Ti_{0.5(1-y)}Al_{0.5(1-y)}Y_y$ targets with Y contents of $y = 0, 2, 4,$ and 8 at%. Scanning electron microscopy showed that all coatings had a dense columnar structure and energy dispersive x-ray spectroscopy indicates slightly higher Y and Al contents in the coating than the respective target. Hence the films from the $Ti_{0.49}Al_{0.49}Y_{0.02}, Ti_{0.48}Al_{0.48}Y_{0.04}$ and $Ti_{0.46}Al_{0.46}Y_{0.08}$ targets were found to contain 3, 5 and 9 % of YN and 56, 52 and 53 % AlN, respectively. Investigations by x-ray diffraction show that the structure of our films changes from single-phase cubic to dual-phase cubic-hexagonal by increasing the Y content from 0 to 3 and 5 % YN, respectively. Coatings containing 9 % YN show a single-phase hexagonal structure. These results were compared to *ab initio* calculations of the energy of formation E_f of cubic and hexagonal $Ti_{1-x}Al_xN$ in dependence of the AlN mole fraction and the Y content. The data show that for AlN fractions below ~ 0.67 the cubic modification is preferred over the hexagonal but this value decreases to ~ 0.5 if 6 at% Y is added. The calculations furthermore indicate an increased specific volume of $\sim 35\%$ of the hexagonal structure with 6 at% Y compared to the cubic $Ti_{0.5}Al_{0.5}N$ structure. This result is in agreement to the obtained thickness increase by $\sim 35\%$ of our films with increasing YN content from 0 to 9 % and the corresponding change in structure from single-cubic to single-hexagonal, respectively.

The intrinsic compressive stresses of our films decrease from -1.8 to -0.7 GPa with increasing YN content from 0 to 9 %, respectively. Corresponding to the change in microstructure from cubic to hexagonal and the decreasing compressive stresses with increasing YN content from 0 to 9 % the hardness and elastic modulus of our films decrease from 33 to 20 GPa and 437 to 196 GPa, respectively.

Stress in WC-DLC Multilayer Coatings

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Compressive stress in tungsten carbide-diamond like carbon (WC-DLC) multilayer coatings has been studied by substrate curvature and transmission electron microscopy. WC-DLC multilayer coatings have been prepared by sputter deposition from a tungsten-carbide target and a time-modulation of the reactive acetylene gas flow. By varying the time-modulation of the acetylene gas flow, the thickness of the individual layers can be varied. It has been observed that compressive stress of the multilayers decrease when reducing the bilayer thickness. Our results show a minimum compressive stress for bilayer thickness of around 5 nm. This behaviour is discussed in terms of interface stress and mixing between layers.

Coating Design by Means of Thermodynamic & TG-Mass Spectrometry Approach

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Thermodynamic approach using computational methods are used in order to predict solid and volatile species of coatings and substrates in contact with corrosive-oxidant environments. Equilibrium condition phases can determine and give a first approach of the protective or non protective character of the coating in relationship with its chemical composition. Also, mass spectrometry technique was used to study in situ the volatile products formation in the oxidation at high temperatures of samples some ferritic steels with slurry and CVD coatings. Approximation of coating design for materials operating in supercritical steam conditions turbines will be given.

Keywords: *mass spectrometry, volatile products, oxidation kinetics, water steam, ferritic martensitic steels, coatings.thermodynami*

Friction properties of ta-C and a-C:H coatings under high vacuum

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The tribological behaviour of ta-C and a-C:H carbon films was analysed by pin-disc tests under high vacuum ($<10^{-3}$ Pa). The tests were executed on a commercial High Vacuum Tribometer (CSM-Instruments) and compared with results obtained under controlled atmospheric pressure conditions. In this work, we present the comparison of the friction coefficients under high vacuum and under controlled atmosphere conditions of two sets of carbon films, one prepared by Filtered Cathodic Vacuum Arc (FCVA; giving rise to hydrogen-free carbon), the other by Plasma Enhanced Chemical Vapour Deposition (PECVD; leading to hydrogenated carbon). The structure of these films was analysed by Raman and Rutherford BackScattering (RBS) spectroscopy and Elastic Recoil Detection (ERD) as well as by X-rays reflectometry (XRR). We will demonstrate that under vacuum, FCVA carbon layers do not slide ($\mu=0.5-0.7$) but PECVD carbon films can have a good sliding behaviour ($\mu=0.1-0.3$). An inverse behaviour was observed under controlled atmospheric conditions. These first results indicate a predominant role of the structure of the films, as analysed by Raman and RBS-ERD spectroscopy as well as by XRR, on the tribological characteristics of the carbon films. In particular, the influence of hydrogen concentration seems to be directly linked to the vacuum tribological behaviour of the coatings.



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Pulsed Power for Pulsed Plasmas: A Physicist's Approach to Highly Ionized Plasmas

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For film synthesis by energetic condensation, it is necessary to produce plasmas with a high degree of ionization. Of particular interest is the ionization of the condensing species, such as metal ions, because this allows us to control the particle energy distribution with electrical fields (bias), and the particle spatial distribution with magnetic fields. This has great consequences for film density, stress, texture, interface engineering, as well as film uniformity.

The most straightforward approach towards a high degree of ionization is to increase the power density by orders of magnitude beyond "typical" discharge power, thereby shifting the electron distribution function and enhancing the probability of ionizing collisions. This is well established in pulsed power physics, and has been studied for decades in z-pinches, wire-explosions, laser ablation plasmas, pseudo-sparks, cathodic arcs, etc. About a decade ago, the concept has been transferred to magnetron sputtering, leading to high power impulse sputtering (HIPIMS). Following the path of a sputtered atom, we recognize that the mean free path between inelastic collisions must be short compared to the characteristic length of the magnetic field structure that keeps the electrons in a closed drift region. This criterion implies a high density of to-be-ionized atoms as well as a significant population of energetic electrons. The realization of both is accomplished by dissipating power in a small volume: the rate of sputtering increases, enabling self-sputtering and enhancing the density of to-be-ionized metal atoms, and the energy of electrons is enhanced, too. Due to the high power load on the metal target, the very high power density can only be sustained in a transient manner, leading to the pulsed nature of this technology.

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Deposition of Transparent Conductive Oxides by High Power Pulsed Magnetron Sputtering (HPPMS)

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High power pulsed magnetron sputtering (HPPMS) has been used for the deposition of ITO films from a ceramic target and Al-doped ZnO films from a metallic target in a reactive process.

ITO films were sputtered at room temperature and 300°C. The film properties were compared to properties obtained by conventional DC sputtering. For both processes the film properties depend

on the amount of oxygen added to the sputtering gas. In case of HPPMS the film properties also strongly depend on the peak power density determined by the charge voltage.

For low temperature deposition the HPPMS process proved to be superior in terms of resistivity obtained for the highest charge voltages used. At 300°C state-of-the-art ITO films are obtained by DC sputtering for optimized oxygen flow using a shielding.

Without such a shielding, undesired spike formation on the film surface was observed. In case of HPPMS it was possible to obtain smooth films with a lowest resistivity of 135 $\mu\Omega\text{cm}$ even without the shielding.

For the reactive deposition of ZnO:Al a new type of process control has been developed in order to stabilize the discharge in the transition region. The discharge characteristics like peak power density and plasma impedance have been analyzed. Films have been deposited at room temperature and 200°C. Resistivities below 400 $\mu\Omega\text{cm}$ have been obtained.

High Power Impulse Magnetron Sputtering Plasmas - A New Tool for Interface Engineering

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An excellent adhesion of hard coatings to steel substrates is paramount in practically all application areas. Conventional methods utilise Ar glow etching or arc discharge pre-treatments that have the disadvantage of producing weak interfaces or adding droplets respectively.

A new tool for interface engineering is high power impulse magnetron sputtering (HIPIMS), [1, 2]. HIPIMS is based on conventional sputtering with extremely high peak power densities reaching 3 kWcm^{-2} at current densities of $>2 \text{ Acm}^{-2}$. HIPIMS of Cr and Nb was used to prepare interfaces on 304 stainless steel and M2 high speed steel (HSS). During the pre-treatment, the substrates were biased to -600 V and -1000 V in the environment of a HIPIMS plasma. The bombarding flux density reached peak values of $>100 \text{ mAcmm}^{-2}$ and consisted of highly ionised metal plasma containing a high proportion of Cr^{1+} and Nb^{1+} but also doubly charged metal ions Cr^{2+} and Nb^{2+} as observed by optical emission and mass spectroscopy. The adhesion was evaluated for a coating consisting of a $0.5 \text{ }\mu\text{m}$ thick CrN base layer and a $2.5 \text{ }\mu\text{m}$ thick nanoscale multilayer stack of CrN/NbN with bilayer thickness of 3.4 nm with typical hardness of $\text{HK}_{0.025} = 3100$ and residual stress of -1.8 GPa . In the case of Cr HIPIMS pre-treatment, the adhesion values on M2 HSS reached scratch test critical load values of $L_C = 70 \text{ N}$ thus comparing well to $L_C = 51 \text{ N}$ for interfaces pre-treated by arc discharge plasmas. Cross-sectional transmission electron microscopy studies revealed a clean interface and large areas of epitaxial growth. The orientation of the coating grains was observed to change together with the orientation of the grains in the polycrystalline steel substrate. Due to the droplet-free pre-treatment by HIPIMS, large scale defects in the coating are minimised and the protection capabilities of the coating against wear and corrosion are enhanced.

[1] W.-D. Münz, A.P. Ehasarian, P.Eh. Hovsepian, *EP 02 011 204.1 (2001)*.

[2] A.P. Ehasarian, P.Eh. Hovsepian, W.-D. Münz, *US 10718435, (2005)*.

High-Power Pulsed Sputtering of Metallic Films with a Newly Designed Magnetron

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High-power pulsed dc magnetron discharges for ionized high-rate sputtering of metallic films were systematically investigated. The depositions were performed using unbalanced circular magnetrons of newly designed types with a directly water-cooled planar copper or titanium target of 100mm in diameter. The magnetron was driven by a pulsed dc power supply working in the frequency range from 0.5 to 50kHz with a maximum pulse voltage and current of 1kV and 120A, respectively. Time evolutions of the magnetron voltage, the target current density (up to 1.5Acm^{-2}) and of the ion current density on the substrate (up to 0.45Acm^{-2} at the distance of 100mm) were measured to provide information on absorption of energy in the discharge plasma and on transfer of arising ions to the substrate. Time-averaged mass spectroscopy was performed at the substrate position to characterize ion energy distributions and composition of the total ion fluxes onto the substrate over a wide range of conditions. Trends in the measured values of the deposition rate per target power density and the ionized fraction of sputtered atoms in the flux onto the substrate were explained on the basis of model predictions.

Oxidation Behaviour of Nanoscale Multilayer CrAlYN/CrN Coatings Deposited by the Combined High Power Impulse Magnetron Sputtering / Unbalanced Magnetron Sputtering, (HIPIMS/UBM) Technique

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A 5 μm thick nanoscale multilayer CrAlYN/CrN coating was produced using an industrial-sized HAUZER HTC 1000-4 system. The surface was pre-treated by intensive bombardment with Ar^+ - Cr^+ ions generated by High Power Impulse Magnetron Sputtering (HIPIMS), [1, 2]. HIPIMS was maintained on 200 x 600 mm target of 99.9 % pure Cr. The CrAlYN/CrN superlattice coating, (superlattice period, $\lambda = 4 \text{ nm}$) was deposited by unbalanced magnetron sputtering using two CrAl targets, one CrAlY and one Cr target. Cr, Al and Y were combined as elements producing dense oxide scales with the aim to develop a PVD coating with enhanced oxidation resistance. The HIPIMS substrate pre-treatment provided for high adhesion of the coating due to promotion of local epitaxial growth over large areas on the coated surface. Critical load values as high as $L_c = 62 \text{ N}$ have been measured in scratch adhesion tests.

The coatings were deposited on polished coupons of 304 stainless steel (SS), M2 high speed steel (HSS) and cemented carbide (CC) substrates for mechanical, tribological and high temperature oxidation characterisation.

The high temperature behaviour of CrAlYN/CrN coatings in air was studied using thermogravimetric analysis (TGA), X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

TGA identified the onset of rapid oxidation of the CrAlYN/CrN deposited on SS to be higher than 950°C . The weight gain after continuous heating up to 1000°C was negligible of 0.5 gm^{-2} .

Isothermal heat treatment in air was carried out at 700, 850 and 1000°C for up to 5 h. XRD analysis revealed that at 1000°C the oxidation process starts with development of a thin Al-rich oxide layer after heat treatment for 30 min.

CrAlYN/CrN was deposited on 8 mm diameter ball nose end mills. Dry high speed ($V_c = 385 \text{ m/min}$) milling test were carried out to machine hardened A2, (HRC=58) high speed steel using MAZAK FJV- 25 milling centre. In this conditions CrAlYN/CrN coated end mills showed a life time of 47 min compared to 39 min achieved with TiAlCrYN and 31 min achieved with TiAlSiN coatings.

[1] W.-D. Münz, A.P. Ehasarian, P.Eh. Hovsepian, EP 02 011 204.1 (2001).

[2] A.P. Ehasarian, P.Eh. Hovsepian, W.-D. Münz, US 10718435, (2005).

Fundamental Behavior of Ions and Arcs in High Power Impulse Magnetron Sputtering

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The plasma in High Power Impulse Magnetron Sputtering (HIPIMS) has been studied using mass spectrometry. It has been found that the HIPIMS discharge contains a much larger fraction of highly energetic ions ($\sim 50\%$ with $E_i > 20$ eV) compared to the continuous DC discharge. The composition of the ion flux revealed a high fraction of target material. During the most intense stage of the discharge the ionic flux contained approximately 50 % Ti^{1+} , 24 % Ti^{2+} , 23 % Ar^{1+} and 3 % Ar^{2+} . Time resolved studies show broad ion energy distributions in the initial phase of the discharge, which rapidly are narrowed down after the pulse switch-off. The origin to this behavior is discussed. Also the formation of arcs in the HIPIMS has been investigated using a number of different materials. It showed the impurity content of the target is a key issue. The relation between arcing frequency and different physical parameters such as melting temperature and cohesive energy is also presented.

First Steps in Time Resolved Optical Emission Spectroscopy of HPPMS Plasma

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In recent years high pulse power magnetron discharges (HPPMS or HIPIMS) more and more becomes a tool for producing dense and pinhole free thin films with better mechanical and corrosion behaviour than it can be achieved with conventional magnetron sputtering, especially on heat sensitive substrates. Particular experimental data had been published concerning the ionization degree of the discharge in the last years. Detailed time-resolved investigations of the optical emission from HIPIMS discharge are relatively rare. The current paper gives an overview about time-resolved OES investigations carried out on different targets with two types of plasma pulsers (Advanced Energy, MELEC). The spectrometer used for these investigations allows different modes of data acquisition: single wavelength pulse mode, multi wavelength time tracking mode as well as scanning mode. The time resolution in pulse mode is 200ns. The spectral resolution ranges from 0.5nm in the VIS down to 0.05nm in the UV region. The investigations presented here include time-resolved spectral scanning measurements (pulse-time spectrum) and multi-wavelength time-resolved measurements. A consideration on the excitation process during the plasma pulse is given. A special section of the paper describes the working principle of the spectrometers solid-state monochromator and its capabilities for time-resolved 2D imaging spectroscopy.

Second-Generation Industrial Sized Power Supply for HIPIMS

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First generation of industrial sized power supply for High Power Impulse Magnetron Sputtering has been presented on the HIPIMS-ABS Days in 2004. It's parameters allowed to drive small to medium sized industrial cathodes (6MW pulse power). Bigger cathodes require more impulse and average power. Additionally improvement of other parameters also has been requested, i.e. pulsing frequency, pulse length. Basing on experience over 2 years tests of the HIPIMS power supply, second generation of the HPM power supply has been developed. It introduces a modular design, what allows to manufacture power supplies in the range of 2-10 MW pulse power. Also the average power may be adjusted to the application (10-60kW). Time resolving parameters have been extended to match deferent cathode materials. Arc management and active arc suppression, even satisfactory in the first generation unit, have been optimized, detection and reaction times further decreased. Communication interface has been changed to use standard component of MP family, what allows to use Analog, Profibus, Bitbus or Internet interface cards.

Experimental results of driving a industrial cathode will be presented on the conference.



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EXHIBITORS

HIPIMS-ABS 2006, Sheffield



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