Advanced Surface Engineering Division
Room 317 - Session SE+MN+PS+TF-WeA

Vapor Deposition Technologies and New Trends in Surface Engineering

Moderators: Jianliang Lin, Southwest Research Institute, **Filippo Mangolini**, The University of Texas at Austin

2:20pm **SE+MN+PS+TF-WeA-1 Breaking the Back-Attraction by Bipolar HiPIMS Bursts***, **Rajesh Ganesan**,* University of Illinois at Urbana-Champaign **INVITED**

Limiting the back-attraction of ions is crucial to increase the deposition rate in HiPIMS processing. Back-attraction can be considerably limited by bipolar plasma bursts in which a positive voltage pulse is applied instantaneously after the negative voltage pulse. Energy-resolved mass spectroscopy confirms that, in addition to the increased flux, the energy of the target metal ions travelling from the target to the substrate is also increased, as a function of positive pulse length. Amorphous carbon coatings have been deposited by bipolar HiPIMS (BiPIMS) as a case study. The increased energy of the depositing flux led to a higher density of the carbon coatings and a significant reduction in the incorporation of the sputter gas atom, argon, was observed in the coatings. Langmuir probe measurements suggest the optimum plasma density window to minimize arc generation and reduce the probability of generated arcs moving away from the target racetrack, which results in smoother coatings. BiPIMS voltage pulses of optimized length and magnitude help to coat high quality amorphous carbon coatings with excellent machining functionalities.

3:00pm **SE+MN+PS+TF-WeA-3 Experimental and Theoretical Study of the Thermal Shock Behavior of MAX Phase Thin Films***, **Matej Fekete**, C. Azina, P. Ondračka, L. Löfler, D. Bogdanovski,* RWTH Aachen University, Germany*; D. Primetzhofer,* Uppsala University, Sweden*; M. Hans, J. Schneider,* RWTH Aachen University, Germany

Components subjected to rapid temperature changes are prone to thermal shock, which may result in damage or catastrophic failure. Thus, thermal shock resistance is one of the performance-defining properties for an application where extreme temperature gradients are required. The thermal shock resistance can be described by the thermal shock parameter (R_T), which depends on the flexural strength, thermal conductivity, Poisson's ratio, linear coefficient of thermal expansion, and elastic modulus. In this study, these thermomechanical properties of Ti_3AlC_2 and Cr_2AlC MAX phase coatings are investigated by both experiment and theory. The R_T of Ti_3AlC_2 obtained through quantum mechanical predictions is in good agreement with the experimentally obtained R_T. However, for Cr_2AlC, the theoretical predictions result in approximately two times larger R_T than experiments. This difference may be caused by omitted spin-polarization in the calculation of the electronic part of the thermal conductivity. Correlating the studied MAX phase thin films, both experiments and theory indicate superior facture behavior of Ti_3AlC_2 in comparison to Cr_2AlC. This is attributed primarily to the higher thermal conductivity of Ti_3AlC_2.

4:20pm **SE+MN+PS+TF-WeA-7 Combinatorial Application of Advanced Characterization Methods to Illuminate the Role of Interfaces in Multilayer Coatings***, **Nina Schalk**, C. Kainz, F. Frank,* Montanuniversität Leoben, Austria*; C. Czettl, M. Pohler,* CERATIZIT Austria GmbH, Austria*; M. Tkadletz,* Montanuniversität Leoben, Austria **INVITED**

The microstructural characterization of multilayer coatings and their interfaces is challenging, especially if the layer thicknesses are only in the nm range. Within this talk, two model coatings are used to evaluate the suitability of several characterization methods for the investigation of their microstructure and interfaces on different length scales. The fine grained cathodic arc evaporated ZrN/TiAlN and the rather coarse grained chemical vapor deposited TiCN/TiC multilayer model coatings exhibit different bilayer thicknesses and layer thickness ratios and thus allow also insight into the effect of the layer thickness on coherency, grain size and strain state. Starting with methods such as scanning electron microscopy and laboratory X-ray diffraction, an overview of the coating structure and information on the average strain/stress state can be obtained. Depending on the grain size and individual layer thickness, high resolution electron backscatter diffraction allows a more detailed insight into the microstructure and strain state of individual layers. In addition, information about gradients of strain/stress across the coating thickness is accessible by cross-sectional X-ray nanodiffraction. However, for a detailed investigation of the interfaces, the application of high resolution methods such as transmission electron microscopy and atom probe tomography is indispensable, providing information about lattice misfits and related strain evolution in the layers as well as on the sharpness of the interfaces in terms of elemental distribution down to the atomic scale. The present talk highlights that for the characterization of the different multilayer systems the combinatorial application of different characterization methods is possible and reasonable.

5:00pm **SE+MN+PS+TF-WeA-9 Influence of Al-Content on Structure, Mechanical Properties and Thermal Stability of Reactively Sputtered AlTaTiVZr High-Entropy Nitride Coatings***, **Alexander Kirnbauer**[1],* TU Wien, Austria*; S. Kolozsvári,* Plansee Composite Materials GmbH, Germany*; P. Mayrhofer,* TU Wien, Austria

In the field of materials research, a novel alloying concept, so-called high-entropy alloys (HEAs), has gained particular attention within the last decade. These alloys contain 5 or more elements in equiatomic or near-equiatomic composition. Properties, like hardness, strength, and toughness can be attributed to the specific elemental distribution and are often superior to those of conventional alloys. In parallel to HEAs also high-entropy ceramics (HECs) moved into in the focus of research. These consist of a solid solution of 5 or more binary nitrides, carbides, oxides, or borides. Within this work, we investigate the structure and, mechanical properties of thin films based on the high-entropy concept, with particular emphasis on the thermal stability, dependent on the Al content in AlTaTiVZr thin films.

Therefore, AlTaTiVZr nitride coatings were reactively sputtered in a lab-scale sputter deposition facility using a single powder-metallurgically produced composite target and Al cubes placed along the racetrack to increase the Al content within the coatings. The coatings in as-deposited state show a fine-columnar growth and crystallise in a single-phase face-centred cubic (fcc) structure. The hardness of our coatings in as-deposited state is ~32.8 GPa and relatively independent on the Al-content. We studied the influence of the Al content on the thermal stability by investigating the structural evolution of our coatings by DSC and powder X-ray diffraction, as well as nanoindentation upon vacuum annealing. The study reveals a distinct influence of the Al-content on the decomposition of the solid solution into an fcc-matrix and Al-rich domains.

5:20pm **SE+MN+PS+TF-WeA-10 Ternary Transition Metal Diborides – Future Defect Engineered Protective Coating Materials?***, A. Hirle, L. Zauner, C. Fuger, A. Bahr, R. Hahn, T. Wojcik, T. Glechner,* Christian Doppler Laboratory for Surface Engineering of high-performance Components, TU Wien, Austria*; J. Ramm, O. Hunold,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein*; P. Polcik,* Plansee Composite Materials GmbH, Germany*; **Helmut Riedl**,* TU Wien, Austria

In the progression of novel protective thin film materials, the attention for transition metal diborides (TMB_2) substantially increased during the last years. The unique strength of their hybridized covalent bonds combined with their hexagonal close-packed (hcp) structures is a big advantage and limiting factor at the same time. The related brittleness, variety of crystal structures, and stoichiometries depict significant challenges for a broad usage of these structurally imperfect coating materials. Furthermore, the formation of non-adherent and volatile oxide scales is also a major limiting factor.

Within this study, we want to address these specific challenges on various ternary model systems within group IV to VI transition metal diborides (e.g. $TM_{1-y}X_yB_{2\pm z}$ prototypes). As structural defects play a major role for the phase formation of the two characteristic hexagonal structure types (α–AlB_2 vs. ω-W_2B_{5-x}-prototype), the target composition and ionization degree within the plasma, has been systematically correlated with the deposition parameters for non-reactive DCMS and HiPIMS depositions. In addition, different alloying concepts for enhancing the ductile character – by microstructural design of imperfect grain boundary structures [1, 2] – as well as oxidation resistance – up to 1200 °C through Si alloying [3] – of these superhard ternary diborides will be discussed in detail. To describe all these relations comprehensively, we correlated the synthesis parameters with structural and morphological evolution using XRD, HR-TEM, APT, as well as micro-mechanical testing methods. Furthermore, specific aspects have also been described by atomistic modelling (DFT).

Keywords : Ternary Borides; Protective Coatings; Defect Engineering; High Temperature Oxidation;

[1] **2021 ASED Young Investigator Awardee**

[1]T. Glechner, H.G. Oemer, T. Wojcik, M. Weiss, A. Limbeck, J. Ramm, P. Polcik, H. Riedl, Influence of Si on the oxidation behavior of TM-Si-B2±z coatings (TM = Ti, Cr, Hf, Ta, W), Surf. Coat. Technol. 434 (2022) 128178.

[2] C. Fuger, R. Hahn, L. Zauner, T. Wojcik, M. Weiss, A. Limbeck, O. Hunold, P. Polcik, H. Riedl, Anisotropic super-hardness of hexagonal WB2±z thin films, Materials Research Letters. 10 (2022) 70–77.

5:40pm **SE+MN+PS+TF-WeA-11 Influence of Interplay of Substrate Template Effects and Bias Voltage on the Microstructure of Cathodic Arc Evaporated Fcc-$Ti_{0.5}Al_{0.5}N$ Coatings**, ***Michael Tkadletz****, N. Schalk, H. Waldl,* Montanuniversität Leoben, Austria*; B. Sartory, J. Wosik,* Materials Center Leoben Forschung GmbH, Austria*; C. Czettl, M. Pohler,* CERATIZIT Austria GmbH, Austria

Ever since the implementation of hard coatings as wear protection for cutting tools, their microstructural design has been of major interest. While the effect of the deposition parameters, such as the applied bias voltage or the substrate temperature, on the microstructure are frequently investigated and rather well understood, commonly less attention is paid to the used cemented carbide substrates. Yet properties like their phase composition and carbide grain size significantly influence the resulting coating microstructure. Thus, within this work substrate template effects are studied on fcc-$Ti_{0.5}Al_{0.5}N$ coatings grown by cathodic arc evaporation onto cemented carbide substrates with different WC grain sizes. A systematic variation of the bias voltage resulted in coarse, intermediate and fine grained coating microstructures, which revealed substrate template-based coating growth at low bias voltages and bias dominated coating growth at high bias voltages. In addition, a strong influence of the applied bias voltage on the resulting preferred orientation of the deposited coatings was observed, providing the basis to tailor the texture to 100, 110 or 111. Elaborate X-ray diffraction and electron microscopy studies contributed to gain further understanding of the substrate template effects and revealed that implementation of a suitable baselayer offers the possibility to effectively prevent any influence of the used substrate on the microstructural evolution of the coating. Supplementary micromechanical experiments illuminated the impact of microstructure, template and non-template based coating growth on the obtained mechanical properties. The obtained results set the fundament to implement tailored microstructures with designed gradients of crystallite size, preferred orientation and consequently mechanical properties, which, as required, either utilize substrate template effects or avoid them.

6:00pm **SE+MN+PS+TF-WeA-12 Super Hard High Temperature TaC-Based Superlattice Protective Coatings Prepared by Magnetron Sputtering**, ***Barbara Schmid****,* TU Wien, Austria*; S. Kolozsvari,* Plansee Composite Materials GmbH, Germany*; P. Mayrhofer,* TU Wien, Austria

Transition metal carbides belong to ultra-high temperature ceramics (UHTC) and are particularly valued for their high thermal and mechanical stability as well as melting points of even above 4000 °C. Therefore, those materials are especially interesting for the application as protective coatings. However, a considerable limitation of these materials is their high inherent brittleness. Inspired by the success of nanolayered superlattice architecture—shown to enhance both hardness and toughness of transition metal nitrides like TiN/CrN or TiN/WN—we developed superlattice films based on TaC. These combinations are motivated by ab initio density functional theory calculations exhibiting large and small shear modulus and lattice parameter misfits. Our coatings are prepared via non-reactive DC and pulsed DC magnetron sputtering using binary carbide compound targets. In our study, we want to compare TaC-based superlattice systems and investigate the influence of the superlattice architecture on material characteristics like mechanical, thermal and electrical properties. Apart from nanoindentation and micromechanical cantilever testing for hardness and fracture toughness, material stability at elevated temperatures as well as thermoelectrical properties are being characterized.

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