Hard Coatings and Vapor Deposition Technologies
Room Town & Country C - Session B5-2-WeA

Hard and Multifunctional Nanostructured Coatings II

Moderators: Rainer Hahn, TU Wien, Institute of Materials Science and Technology, Austria, **Tomas Kozak**, University of West Bohemia, Czechia

2:00pm **B5-2-WeA-1 Design of Coatings for Harsh Environments by Computation and Experiment (Virtual Presentation)**, ***Efstathios "Stathis" I. Meletis (meletis@uta.edu)***, University of Texas at Arlington, USA **INVITED**

One of the great challenges in engineering science is to protect a material at its surface from chemical reactions and mechanical degradation such as high temperature oxidation, impact and wear. Applying a hard coating, which bonds strongly to the surface of the material, prevents excessive abrasion and provides the needed shield towards mechanical impact. At high temperatures in an oxidative environment, however, many hard boride, carbide and nitride coatings quickly deteriorate due to thermal instability and chemical degradation. An overview is presented of our recent efforts under the materials genome initiative to develop a new class of protective ceramic coatings, coalescing computational investigation and experimental realization and characterization. The efforts focus on several transition metal quaternary (Zr,Hf,Si)BCN and ternary (Hf,Ta)SiN amorphous and nanocomposite coatings for severe environment applications. Compositional and structural atomistic simulations using density-functional theory and large-scale molecular dynamic calculations were conducted to explore thermal, oxidation and mechanical properties. A number of complementary experimental characterization techniques were used to study the thermal, mechanical and oxidation resistance of the coatings. Atomistic and local structure characterization and image simulations were conducted by using HRTEM to develop a comprehensive understanding of the synthesis-structure-property relationship in these high potential coating systems.

2:40pm **B5-2-WeA-3 Microstructure and Properties of PVD Synthesized Super-hard Ti-B-N Coatings**, ***Rebecca Janknecht (rebecca.janknecht@tuwien.ac.at)**, R. Hahn, A. Kirnbauer,* TU Wien, Institute of Materials Science and Technology, Austria; *P. Polcik* , Plansee Composite Materials GmbH, Germany; *P. Mayrhofer,* TU Wien, Institute of Materials Science and Technology, Austria

Ternary transition-metal boron nitride Ti-B-N has emerged as an outstanding material system in the field of protective coatings due to its high hardness and temperature stability. In particular, its large number of equilibrium and metastable phases offers numerous possible compositions for investigating and understanding structural effects and their influence on mechanical properties.

With prior interest in chemical compositions close to the TiN-TiB line in the corresponding equilateral concentration triangle, Ti-B-N coatings were prepared using a Ti-TiN-TiB_2 target containing 10 at.% B.

Focusing on the investigation of single-phase structured super-hard (H≥40 GPa) Ti-B-N coatings, the influence of various deposition conditions on the structure and mechanical properties during non-reactive DC magnetron sputtering - compared to nanocrystalline Ti-B-N - comprises this study. The results show - consistent with previous ab initio calculations - that up to 10 at.% B can be incorporated into the face-centered cubic (fcc) TiN lattice.

3:00pm **B5-2-WeA-4 Enhanced Mechanical Performance of Nanostructured B-Dopednitride Coatings Deposited by HiPIMS With Positive Pulses**, *P. Diaz-Rodriguez, A. Mendez, J. Santiago,* ***Ivan Fernandez (ivan.fernandez@nano4energy.eu)**, A. Wennberg, J. Endrino,* Nano4Energy, Spain; *E. Chacon, A. Guzman, M. Panizo,* Universidad Politecnica de Madrid, Spain; *M. Monclus, J. Molina,* IMDEA Materiales, Spain

In recent years, due to the advancement of high-speed machining(HSM), more demanding specifications on cutting tool coatings' hardness, chemical inertness materials, wear resistance,anti-abrasion, and also thermal and oxidation resistance are required. In order to overcome the detrimental effects associated with high temperatures during HSM on tool life and workpiece surface finishing, nanostructured coatings based on multilayers or nanocomposites have been proposed [1, 2]. In this work, we present nanostructured AlTiBN and AlCrBN coatings deposited by HiPIMS with positive pulses. The optimization of the coatings was carried out by tailoring metal ion fluxes and energies. More energetic process conditions have been provided by adjusting height and width of positive pulses. Coatings' microstructure has been studied and related to HiPIMS parameters. The formation of nanocrystalline grains embedded in an amorphous boron-rich phase provides enhanced toughness and wear resistance[3]. Hardness up to 40 GPa were measured by nanoindentation techniques and high adhesion critical load values were obtained in nanoscratch testing. High temperature nanoindentation and micropillar splittingwere used to evaluate toughness and thermal resistance of the coatings. Finally, micromilling tests were carried out to assess the performance of these nanostructured coatings in micromachining of stainless steel and titanium alloys.

[1] J. Musil,Surface and Coatings Technology 125 (2000) 322–330

[2]P. Mayrhofer et al.,Progress in Materials Science 51 (2006) 1032–1114

[3] A.Mendez et al., Surface and Coatings Technology 422 (2021) 127513

3:20pm **B5-2-WeA-5 Development of TiB_2 Coatings in a New Generation Industrial Reactor Based on Hybrid DC-pulsed and HIPIMS Magnetron Sputtering on HSS Steels – Tribological Study at Room, Medium and High Temperature**, *E. Arias,* Asociación de la Industria Navarra, Spain; ***Gonzalo Garcia fuentes (gfuentes@ain.es)***, Asociación de la industria Navarra, Spain; *H. Gabriel,* PVT Plasma und Vakuum Technik GmbH, Germany; *I. Fernández,* N4E, Spain; *J. Fernández Palacio,* Asociación de la Industria Navarra, Spain

Titanium di-boride (TiB_2) coatings exhibit excellent combination of hardness and low adhesion to cutting metal alloys such as these based on Ti, Al or Ni, and it has been used since a decade on cutting tools in the aerospace sector. TiB_2 is well known to exhibit low moderate toughness, which limits it applicability under complex 3D shaped cutting tools, or tools subject to very high loads. Pulsed DC sputtering as well as other conventional vapor deposition techniques are being developed to this purpose. In our approach, a hybrid industrial scale system equipped with 4 magnetron sputtering sources and a 600/350 mm Height/Diameter effective volume is chosen to implement TiB2 coating formulations. The system is equipped with pulsed DC and HIPIMS V+ PSUs. The target configuration is chosen in the unbalance mode while the BIAS pulse is synchronized with the HIPIMS PSU so to enhance the Me+ ion bombardment ratio. The HIPIMS V+ parameterization will be focus to provide an enhanced coating-to-substrate adhesion strength, but also microstructural strength to the TiB_2 matrix. Optical emission probes will provide valuable information about the concentration ratio of Me/Ar ionized species for different pulsing/power deposition conditions. The characterization of the coatings is carried out using glow discharge emission spectroscopy, x-ray diffraction, scanning electron microscopy and nanoindentation. The friction and wear is characterized using different conditions of temperature, and load. In particular, the surface contact interaction with Ti-alloys and Al-alloys will be discussed in terms of the galling of the testing materials for different conditions.

3:40pm **B5-2-WeA-6 Study the Effect of Nozzle Geometry on Spray Coating by Aerosol Deposition Method**, ***Bahareh Farahani (bahareh.farahani@csulb.edu)***, California State University, Long Beach, USA; *M. Jadidi,* Ryerson University, Canada; *S. Moghtadernejad,* California State University, Long Beach, USA

The Aerosol Deposition (AD) method, also known as Vacuum Cold Spraying (VCS), is an emerging spray coating technology to fabricate a dense thick/thin ceramic or metal layer on a substrate through the kinetic impaction and cumulative deposition of ultrafine solid particles under near-vacuum conditions [1]. AD is a Room Temperature Impact Consolidation (RTIC) mechanism that causes negligible oxidation or degradation to the feedstock powders making it a desirable candidate for applying coatings on lower-melting and temperature-sensitive substrates such as glass, metal, and polymers. This high-velocity deposition technique is a promising coating process for industrial applications, such as solid oxide fuel cells, solid-state lithium batteries, bio-component coatings, and surface protection [1]. One of the challenges of this method is achieving successful deposition of the particles with high coating quality. It should be noted that coating quality can be significantly improved by optimizing the particle impact velocity. As such, the focus of this project is to study the effect of nozzle geometry and operating parameters such as range of pressure ratios and standoff distance on particle impact velocity using the method of Computational Fluid Dynamics (CFD).

In this work, a de Laval nozzle with two axisymmetric geometries of 1) sharp and 2) rounded edge will be modeled to generate a supersonic gas flow to accelerate particles toward a substrate using CFD. To simulate the gas flow behavior in the jet stream leaving the nozzle, a realizable k-epsilon turbulence model will be used. Results will be validated by comparing the locations of the Mach disks in highly under-expanded free-jet conditions with the experimental data of nozzles with the same geometries. In addition, a Eulerian-Lagrangian based discrete-phase model will be used to study the particle trajectory and velocity upon impact with the substrate. The effect of drag coefficient and thermophoretic force on in-flight particles behavior will be also evaluated to improve the particles deposition formation upon impact. The simulation results will lead to understanding the effects of nozzle geometry and operating parameters on the particle impact velocity and distribution. This ultimately provides the baseline to design and optimize an AD system with enhanced coating quality.

[1] D. Hanft, J. Exner, M. Schubert, T. Stöcker, P. Fuierer, and R. Moos, "An overview of the Aerosol Deposition method: Process fundamentals and new trends in materials applications," *J. Ceram. Sci. Technol.*, vol. 6, no. 3, pp. 147–181, 2015, doi: 10.4416/JCST2015-00018.

4:00pm **B5-2-WeA-7 Thick Ceramic Coatings Deposited by Supercritical Fluid Chemical Deposition (SFCD)***,* ***Erwan Peigney (erwan.peigney@icmcb.cnrs.fr)****, G. Aubert,* ICMCB-CNRS, France*; M. Cavarroc,* SAFRAN, France*; A. Poulon-Quintin, C. Aymonier,* ICMCB-CNRS, France

Requirements for the elaboration of ceramic coatings are constantly evolving, especially when it comes to depositing high thicknesses on complex shaped substrates. In this way, traditional deposition methods do not fully meet the new challenges, notably directional processes such as PVD. Liquid-phase processes suffer also many limitations, including contamination issues and poor mass transport. Likewise, non-directional gas-phase processes such as CVD encounters precursor volatility constraints, which lead to mass transport-limited conditions, poor step coverage and non-uniformity for submicrometer-patterned substrates.

To overcome all these difficulties, new processes have shown great interest in recent decades. Among this, Supercritical Fluid Chemical Deposition technology (SFCD) stands out thanks to its advantages. Indeed, supercritical fluids have hybrid thermophysical properties, intermediate between liquids and gases, which are continuously adjustable with small variations of pressure and temperature. For instance, they exhibit gas-like viscosities and diffusivities while having liquid-like densities allowing the dissolution of a wide range of metallic precursors with substantial concentrations. SFCD process proposes to deposit thick inorganic films with uniform coverage of complex shaped substrate, complete filling of narrow high aspect ratio features, reduction in process temperatures and this with a high growth rate deposition.

The present work concerns the development of thick ceramic coatings on complex shaped metallic substrates using a cold-wall reactor filled with different mixture of fluids at sub- and supercritical conditions. Depending on the precursor selected, the fluid composition and the deposition parameters, the coating properties are tuned in terms of adhesion to the substrate, hardness, roughness and corrosion resistance. The material microstructure and crystallinity are also impacted. They are characterized through GIXRD, TEM, XPS and Raman spectroscopy.

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