

Protective and High-temperature Coatings

Room Golden State Ballroom - Session MA-ThP

Protective and High-temperature Coatings Poster Session

MA-ThP-1 High Temperature Fracture Characteristics of Si Containing Ternary and Quaternary Transition Metal Diborides, Anna Hirle [anna.hirle@tuwien.ac.at], Ahmed Bahr, Rainer Hahn, Tomasz Wojcik, Christian Doppler Laboratory for Surface Engineering of High-performance Components, TU Wien, Austria; Szilard Kolozsvári, Peter Polcik, Plansee Composite Materials GmbH, Germany; Jürgen Ramm, Carmen Jerg, Oerlikon Surface Solutions AG, Liechtenstein; Helmut Riedl, Christian Doppler Laboratory for Surface Engineering of High-performance Components, TU Wien, Austria

To enhance the restricted oxidation resistance of transition metal diboride (TMB) ceramics, alloying with Si and silicide phases is an effective method, resulting in the formation of highly dense and protective SiO₂ scales. This phenomenon has been well documented in the context of bulk ceramics [1, 2], and recent studies have also corroborated its occurrence in thin-film TMBs, including CrB₂, HfB₂, and TiB₂ [3, 4]. The incorporation of Si, TaSi₂ or MoSi₂ into TiB₂ results in a significant reduction in oxidation kinetics, while exhibiting only minor effects on the mechanical properties. In the case of quaternary TiB₂-based coatings, hardness values of 36 GPa (TaSi₂) and 27 GPa (MoSi₂) have been achieved, in comparison to approximately 38 GPa for the binary system. All of the aforementioned coatings exhibited α-AlB₂ crystal structure, with a preferred (0001) orientation being a key factor in achieving the highest hardness. Nevertheless, the fracture characteristics of these Si-alloyed TMBs remain largely unexplored.

The objective of the present study is to elucidate the fracture characteristics, particularly K_{IC}, of these Si-containing TMBs at elevated temperatures up to 850 °C through the application of in-situ micromechanical testing techniques. Accordingly, a series of Ti-TM-Si-B_{2+z} coatings was deposited via non-reactive DC magnetron sputtering using a variety of composite targets, including TiB₂, TiB₂/TiSi₂ (90/10 & 80/20 mol%), TiB₂/TaSi₂ (90/10 & 80/20 mol%), and TiB₂/MoSi₂ (85/15 & 80/20 mol%). To gain a deeper understanding, additional detailed structural investigations were conducted using X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and elastic recoil detection analysis (ERDA).

In comparison to the binary TiB_{2+z} and the quaternary Ti-Ta-Si-B_{2+z}, the Si and MoSi₂-containing coatings exhibited a distinct onset of plastic deformation at approximately 600 °C. This phenomenon can be attributed to the precipitation of silicon-containing phases, which underlines the significance of conducting material testing at temperatures relevant to their intended applications.

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MA-ThP-2 Influence of Si on the Oxidation Behavior of High Entropy Carbide Thin Films Based on (Hf, Ta, Ti, V, Zr)C, Muhammad Awais Altaf [muhammad.altaf@tuwien.ac.at], Alexander Kirnbauer, Balint Hajas, TU Wien, Institute of Materials Science and Technology, Austria; Szilard Kolozsvári, Plansee Composite Materials GmbH, Germany; Paul Mayrhofer, TU Wien, Institute of Materials Science and Technology, Austria

In the present work, the influence of Si addition on the oxidation behavior of high-entropy carbide thin films based on the system(Hf, Ta, Ti, V, Zr)C is investigated. High-entropy carbides thin films were deposited on sapphire (Al₂O₃) as well as low-alloy steel substrates using magnetron sputtering. Additionally, powdered free-standing coating materials were also produced. All the as-deposited thin films exhibit a single-phase face-centered cubic (FCC) structure (Fm-3m, space group number 225). During non-isothermal oxidation using DSC-TGA, onset-temperature shifted from 490 °C to 502 °C while mass gain was reduced from 11% to 8.88 % with Si addition. Furthermore, the oxide-peak intensities in the obtained XRD patterns are decreased indicating a smaller fraction of formed oxide phases. All samples were fully oxidized during isothermal oxidation but the samples alloyed with Si show denser and thinner oxide scales compared to the samples without Si.

MA-ThP-3 Spinodal Decomposition and Nano-precipitate Formation in Ag-modified High-Entropy Alloys, Salah-eddine Benrazzouq [salah-eddine.benrazzouq@univ-lorraine.fr], Abdelkrim Redjaimia, Jaafar Ghanbaja, Sylvie Migot, Valentin A. Milichko, Jean-François Pierson, Institut Jean Lamour - Université de Lorraine, France

Phase separation in multi-component alloys presents both challenges and opportunities for material design. While traditionally viewed as a limitation, controlled phase separation could enable unique microstructural features and enhanced properties. High-entropy alloys (HEAs) have garnered significant attention across various research fields owing to their exceptional properties. This study investigates the distinctive behavior of silver addition to the CrMnFeCoNi Cantor alloy, where silver's higher mixing enthalpy creates an interesting case of spinodal decomposition and nano-precipitate formation.

Using DC magnetron co-sputtering, we synthesized CrMnFeCoNiAg thin films with systematically varied silver content. X-ray diffraction (XRD) patterns reveal distinct non-mixing behavior with the emergence of pronounced peaks corresponding to both silver and Cantor alloy phases. Cross-section bright-field TEM micrograph and SAED patterns revealed a dense structure with Ag precipitates dispersed throughout the 900-nm-thick film. HRTEM micrographs showed a nanoprecipitate morphology with fine-scale linear precipitates, while STEM-HAADF imaging highlighted the internal structure, revealing characteristic modulated patterns with striations parallel to the basal plane, indicative of spinodal decomposition with cuboidal particles and tweed-like contrast patterns.

The controlled formation of these nano-precipitates and their unique distribution pattern suggests potential for mechanical property enhancement through precipitation strengthening mechanisms. Our findings demonstrate how controlled phase separation can be used to engineer microstructure in HEA thin films. This understanding provides new strategies for designing multi-functional materials through deliberate exploitation of immiscibility effects, advancing our knowledge of phase evolution in complex alloy systems and offering pathways for property optimization in advanced coating applications.

MA-ThP-4 Influence of Si Content on Cracking Behavior of CrAlSiN Coatings, Kirsten Bobzin, Christian Kalscheuer, Max Philip Möbius [moebius@iot.rwth-aachen.de], Jessica Borowy, Surface Engineering Institute - RWTH Aachen University, Germany

The increasing demands for workpiece quality and cost-effectiveness in machining processes necessitate a comprehensive consideration of all relevant factors, including cutting parameters, materials, tool coatings, and geometry. Physical Vapor Deposition (PVD) manufactured CrAlSiN nanocomposite coatings, composed of CrAlN grains in a SiN_x matrix, represent a promising solution for improved tool life of milling tools. The elastic-plastic properties of the coating and the deformation behavior of the material composite thereby can be deliberately influenced by varying the silicon content.

CrAlSiN coatings with silicon contents of x_{Si} = 10, 15, 20, and 25 at.-% in the metal portion were fabricated on cemented carbide WC-Co substrates. The indentation hardness H_{IT} and indentation modulus E_{IT} of the coatings were measured through nanoindentation (NI) with a force of F_{NI} = 10 mN, using a Berkovich indenter. Additionally, crack resistance was evaluated using quasi-static high load (HL) nanoindentation tests under forces ranging from F_{HL} = 750 to 1,750 mN, with increments of ΔF_{HL} = 250 mN. A conical diamond indenter was used for the high load nanoindentation tests. The resulting indents were subsequently analyzed using scanning electron microscopy (SEM). The findings reveal that the indentation hardness H_{IT} remains unchanged at H_{IT} = (25.48 ± 1.59) GPa, while the indentation modulus increases with higher silicon content, ranging from E_{IT} = (222.64 ± 10.45) GPa for x_{Si} = 10 at.-% up to E_{IT} = (239.89 ± 7.78) GPa for x_{Si} = 20 at.-%. After high load nanoindentation all coatings exhibit no cracks at F_{HL} = 750 mN. With F_{HL} ≥ 1,000 mN on the other hand cracks can be observed in all coatings. Nevertheless, with rising silicon content, the maximum indentation depth h_{max} decreases, while the residual indentation depth h₀ remains constant. Furthermore, the proportion of plastic work shows a slight reduction as silicon content x_{Si} increases. These results indicate that the resistance against plastic deformation of the CrAlSiN coating increases with higher silicon content.

Coatings with high silicon content demonstrate promising resistance against plastic deformation at room temperature, highlighting their potential for further investigation. This initial test qualifies these coatings for additional studies under high-temperature conditions, aiming to enhance their applicability in machining processes. The insights gained from this research

could lead to the development of more durable and efficient cutting tools, ultimately improving productivity in industrial applications.

MA-ThP-5 Relationship between Optical and Electrical Properties and the Microstructure of High Entropy Nitride (TiVZrNbTa)_xN_x Thin Films, Miguel Piñeiro [miguel.pineiro-sales@univ-lorraine.fr], Institut Jean Lamour - Université de Lorraine, France, Peru; *Salah-Eddine Benrazouq*, Institut Jean Lamour - Université de Lorraine, France, Morocco; *Alexandre Bouché, Valentin Milichko, David Pilloud, Thomas Easwarakhanthan*, Institut Jean Lamour - Université de Lorraine, France; *Frank Mücklich*, Saarland University, Germany; *Jean-François Pierson*, Institut Jean Lamour - Université de Lorraine, France

In this study, high entropy nitride TiVZrNbTa thin films were prepared by DC reactive magnetron sputtering on silicon substrates at room temperature. The impact of varying nitrogen flow rates on the structural, microstructural, optical and electrical properties were investigated. X-ray diffraction technique revealed that all the deposited films exhibited a polycrystalline structure with fcc phase. However, the pure metallic samples displayed an amorphous structure [1]. Optical properties analysis showed a decrement of the reflectance compared with free-nitrogen sample in the infrared region, as determined by UV-VIS spectroscopy [2]. Hall-effect measurements indicate that the electrical resistivity for all samples remained within the range between 100 and 300 $\mu\Omega$ cm. Interestingly, samples deposited with applied substrate bias power during the deposition process did not show a significant change in resistivity. This suggests that substrate biasing has minimal effect on the electrical transport properties of the latter films. On the other hand, applying adjustable substrate bias led to a blueshift in the epsilon-near-zero (ENZ) wavelength. Furthermore, X-ray photoelectron spectroscopy (XPS) shows the effect of the nitrogen flow rate on the residual stress [3] and plasmon frequency. The impact of varying nitrogen flow rates on the microstructural properties were further investigated and explained.

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MA-ThP-6 Microstructure Evolution and Oxidation Behavior of Diffusion Pt- γ/γ' and Pt-aluminide Coatings at 1200 °C, Radoslaw Swadzba [radoslaw.swadzba@git.lukasiewicz.gov.pl], Agnieszka Sasiela, Lukasiewicz Research Network - Uppersilesian Institute of Technology, Poland; *Boguslaw Mendala, Lucjan Swadzba*, Silesian University of Technology, Poland; *Lukasz Pyclik, Michal Gut*, Avio Polska sp. z o. o., Poland This study examines the microstructural evolution and oxide scale growth of Pt- γ/γ' and Pt-aluminide diffusion coatings applied to a second-generation single-crystal Ni-based superalloy at 1200 °C. The Pt- γ/γ' coatings were produced through platinum electroplating followed by a 2-hour diffusion heat treatment at 1079 °C. Subsequently, Vapor Phase Aluminizing (VPA) at 1079 °C for 6 hours generated Pt-modified aluminide coatings. Both coating types were subjected to Thermogravimetric Analysis (TGA) in air for 20 hours as well as cyclic oxidation test up to 300 1-hour cycles at 1200 °C.

The initial and oxidized coatings were characterized using Electron Backscatter Diffraction (EBSD) to analyze phase transformations, grain size evolution, and interdiffusion between the coatings and the substrate alloy. In the as-deposited state, the Pt- γ/γ' coating consisted of γ and γ' grains enriched in Pt, with a thickness of approximately 27 μm , while the Pt-aluminide coating exhibited an outer zone of PtAl₂ and β -NiAl phases. During the high temperature oxidation testing, the Pt- γ/γ' coating showed grain growth and Pt diffusion to a depth of approximately 70 μm after 20 hours. The Pt-aluminide coating underwent martensitic transformation in its outer layer, with Al-depleted β -NiAl in its middle region and an interdiffusion zone containing Cr-rich precipitates.

High-resolution Scanning Transmission Electron Microscopy (STEM) provided detailed characterization of the alumina oxide scales formed on both coatings, revealing information on oxide grain size and the segregation of reactive elements (RE) to grain boundaries.

MA-ThP-7 Unprecedented B Solubility in Cubic (Hf,Ta,Ti,V,Zr)B-C-N Coatings, Andreas Kretschmer, TU Wien, Austria; Marcus Hans, Jochen Schneider, RWTH Aachen University, Germany; Paul Mayrhofer [paul.mayrhofer@tuwien.ac.at], TU Wien, Institute of Materials Science and Technology, Austria

We investigate the influence of compositional complexity in Ti-B-C-N-based coatings by depositing Ti-rich (Hf,Ta,Ti,V,Zr)B-C-N coatings with varying B/C ratios (from 0/21 to 32/0 at%). Despite the high B content of 32 at% in the C-free material, this (Hf_{0.1}Ta_{0.1}Ti_{0.6}V_{0.1}Zr_{0.1})B_{0.6}N_{0.4} forms a single-phase fcc solid solution without a boride tissue phase. All coatings—(Hf_{0.1}Ta_{0.1}Ti_{0.6}V_{0.1}Zr_{0.1})B_xC_{0.5-x}N_{0.5} with $x = 0.2, 0.3, 0.4$, (Hf_{0.1}Ta_{0.1}Ti_{0.6}V_{0.1}Zr_{0.1})B_{0.6}N_{0.4}, and (Hf_{0.1}Ta_{0.1}Ti_{0.6}V_{0.1}Zr_{0.1})C_{0.4}N_{0.6}—exhibit similar hardness values of 37–38 GPa, but increasing B content leads to a decreasing indentation modulus. This trend is supported by *ab initio* calculations of fcc-(Hf_{0.1}Ta_{0.1}Ti_{0.6}V_{0.1}Zr_{0.1})B_xC_{0.5-x}N_{0.5} (for $x = 0, 0.125, 0.25, 0.375, 0.5$), which also confirm the stability of these solid solutions over a wide compositional range. Despite increasing chemical complexity, the addition of B and C has little effect on lattice distortion.

Among the investigated coatings, (Hf_{0.1}Ta_{0.1}Ti_{0.6}V_{0.1}Zr_{0.1})B_{0.4}C_{0.1}N_{0.5} provides the best balance between high hardness (37.7 \pm 1.0 GPa) and fracture toughness ($K_{IC} = 4.0\pm 0.5$ MPa·m^{0.5}). This compositionally complex, single-phase, fcc-structured Ti-rich (Hf,Ta,Ti,V,Zr)B-C-N retains its hardness—which even slightly increases to 38.3 \pm 1.3 GPa—upon vacuum annealing up to 1200 °C. X-ray diffraction and atom probe tomography confirm its high-temperature phase stability, as an hcp-TiB₂-based phase forms only upon annealing beyond 1200 °C. More generally, all (Hf_{0.1}Ta_{0.1}Ti_{0.6}V_{0.1}Zr_{0.1})B_xC_{0.5-x}N_{0.5} coatings with $x = 0.2, 0.3, \text{ and } 0.4$ exhibit a total configurational entropy of ~ 1.1 -R (~ 1.25 -R at the metal sublattice and 0.95-R at the non-metal sublattice) and maintain a hardness of 36–38 GPa even when annealed at 1200 °C, contrary to compositionally simpler coatings, which soften to below 29 GPa.

These findings highlight the advantages of compositionally complex mixed ceramic coatings, which outperform simpler Ti-B-N or Ti-C-N coatings with similar structure and composition. Furthermore, they demonstrate how solubility limits can be extended beyond currently known boundaries through advanced materials science, enabling outstanding properties.

MA-ThP-8 Ab Initio Assessed Influence of Si on the Structural Integrity of Group IV Transition Metal Diborides, Christian Gutschka, Lukas Zauner, Thomas Glechner, Christian Doppler Laboratory for Surface Engineering of high-performance Components, TU Wien, Austria; David Holec, Department of Materials Science, Montanuniversität Leoben, Austria; Helmut Riedl [helmut.riedl@tuwien.ac.at], Institute of Materials Science and Technology, TU Wien, Austria

Transition metal diborides, a class of refractory ceramics, have been shown to exhibit remarkable high-temperature stability and mechanical properties, encouraging research on their bulk and thin film forms. Scientific interest has been directed towards the formation of meta-stable solid solutions with silicon, with the aim of enhance oxidative properties and fracture characteristics. However, theoretical investigations of such ternary compounds remain rare. Therefore, in this study the structural, energetical, and mechanical properties of the Ti-Si-B₂, Zr-Si-B₂, and Hf-Si-B₂, as well as their vacancy dynamics, were explored with the help of Density Functional Theory (DFT). In all three systems, silicon is observed to prefer the boron sublattice. Through structural analysis, solubility limits of 24 at. %, 27 at. %, and 25 at. % of Si in Ti(Si,B)₂, Zr(Si,B)₂, and Hf(Si,B)₂, could be established, respectively. An analysis of simulated XRD patterns, Radial Distribution Functions (RDFs), and Crystal Orbital Hamilton Populations (COHPs), revealed that the loss of AlB₂-type symmetry could be attributed to the formation of Si clusters. Simulations of elastic properties demonstrated a reduction of Young's moduli but enhancing ductility criteria, both with increasing silicon contents, which was in line with experimental values up to 15 at. % Si. Concerning defects, the study revealed a structural instability of ternary AlB₂-type compounds with respect to metal vacancies. Furthermore, it was observed that both metal and boron vacancies showed a decreasing influence on the formation energies as the Si content increased.

MA-ThP-9 Fabrication and High-Temperature Test of Light-Weight Insulation Materials and Coatings for Reusable Thermal Protection Materials, Seongwon Kim [woods3@kicet.re.kr], Korea Institute of Ceramic Engineering and Technology, Republic of Korea Light-weight ceramic insulation materials and high-emissivity coatings were fabricated for reusable thermal protection systems (TPS). Alumina-silica fibers and boric acid were used to fabricate the insulation, which was heat

treated at 1250°C. High-emissivity coating of borosilicate glass modified with TaSi₂, MoSi₂, and SiB₆ was applied via dip-and-spray coating methods and heat-treated at 1100°C. Testing in a high-velocity oxygen fuel environment at temperatures over 1100°C for 120 seconds showed that the rigid structures withstood the flame robustly. The coating effectively infiltrated into the fibers, confirmed by scanning electron microscopy, energy-dispersive X-ray spectroscopy, and X-ray diffraction analyses. Although some oxidation of TaSi₂ occurred, thereby increasing the Ta₂O₅ and SiO₂ phases, no significant phase changes or performance degradation were observed. These results demonstrate the potential of these materials for reusable TPS applications in extreme thermal environments.

MA-ThP-10 Development of Multilayer Nano Nitride Layer for Corrosion and Wear Resistance by Using Magnetron Sputtering Technique, Aakanksha Jain [aakanksha_j.me@iitr.ac.in], India Institute of Technology Roorkee, India; *Ramesh Chandra*, India Institute of Technology Roorkee, India; *Rahul S. Mulik*, India Institute of Technology Roorkee, India

This study investigates the development of multilayer nano nitride coatings for enhanced corrosion and wear resistance, fabricated using the magnetron sputtering technique. The multilayer coatings, consisting of alternating thin nitride layers with tailored stoichiometries and thicknesses, are designed to improve mechanical properties and protect substrates from aggressive environments. The corrosion performance of the coatings was assessed using Electrochemical Impedance Spectroscopy (EIS), a technique that provides valuable insight into the electrochemical behavior and protective efficiency of the coatings in corrosive media. The EIS results demonstrated a marked improvement in the corrosion resistance of the multilayer coatings compared to uncoated substrates and single-layer coatings, indicating their superior ability to act as a barrier against corrosive agents.

Nanoindentation was employed to evaluate the mechanical properties, particularly the hardness of the coatings. This technique allowed for precise hardness measurements at the nanoscale, revealing a significant increase in hardness for the multilayer coatings compared to both the substrate and single-layer nitride coatings. The improved hardness is attributed to the unique microstructure and the stress distribution across the multilayer design, which enhances wear resistance and mechanical durability.

The coatings' microstructure, phase composition, and adhesion strength were further characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and scratch testing, confirming the high quality of the multilayer coatings and their strong interlayer bonding. Overall, the multilayer nitride coatings exhibited enhanced mechanical and electrochemical properties, providing excellent corrosion and wear resistance. These results suggest that magnetron sputtering, combined with EIS and nanoindentation, is an effective approach for developing advanced nitride coatings suitable for applications in harsh industrial environments requiring high durability and long-term performance. This study will help in marine applications for future purposes and be beneficial.

Keywords: Magnetron sputtering, Nitride coatings, hardness, corrosion, marine applic

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