Protective and High-temperature Coatings
Room Palm 3-4 - Session MA1-2-MoA

Coatings for High Temperatures and Harsh Environment Applications II

Moderators: Vladislav Kolarik, Fraunhofer Institute for Chemical Technology ICT, Germany, **Fernando Pedraza**, La Rochelle University, Laboratory LaSIE, France

1:40pm **MA1-2-MoA-1 Synergistic Effects of Ta and Si Alloying on the Longterm Oxidation and Hot Corrosion Resistance of Ti–Al–N Coatings***, **Anna Hirle [anna.hirle@tuwien.ac.at]**, Rainer Hahn, Oliver E. Hudak, Philip Kutrowatz, Tomasz Wojcik,* Christian Doppler Laboratory for Surface Engineering of High-performance Components, TU Wien, Vienna, Austria*; Szilard Kolozsvári, Peter Polcik,* Plansee Composite Materials GmbH, Lechbruck am See, Germany*; Anders.O Eriksson, Carmen Jerg, Klaus Boebel,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Balzers, Liechtenstein*; Helmut Riedl,* Christian Doppler Laboratory for Surface Engineering of High-performance Components, TU Wien, Vienna, Austria; Institute of Materials Science and Technology, TU Wien, Vienna, Austria

$Ti_{1-x}Al_xN$ is one of the most used coating materials applied in various applications, including i.e. machining and forming tools but also components, due to its excellent thermomechanical properties. However, as operating temperatures rise, new strategies are needed. Alloying $Ti_{1-x}Al_xN$ with Ta or Si shows promise. In more detail, the incorporation of Ta shifts the onset of the spinodal decomposition towards higher temperatures. Furthermore, the formation of the unfavoured anatase phase during oxidation is suppressed and the rutile phase is stabilised [1,2]. Adding Si enhances the thermal stability and oxidation resistance, while concomitantly leading to the formation of a nanocomposite microstructure [3]. Recent research explores combined alloying with Ta and Si to improve both oxidation resistance and mechanical performance. [4,5]. Compared with $Ti_{1-x}Al_xN$ coatings – which fully oxidize at 1000 °C (15 h, synthetic air) – $Ti_{1-x-y-z}Al_xTa_ySi_zN$ thin films form oxide scales below 1 µm [5].

Furthermore, $Ti_{1-x-y-z}Al_xTa_ySi_zN$ coatings exhibit excellent mechanical properties, making them promising candidates for high temperature applications [4].

The present study investigates a series of $Ti_{1-x-y-z}Al_xTa_ySi_zN$ coatings deposited by cathodic arc evaporation using an industrial-scale Oerlikon Balzers INNOVA 1.0 system. Two distinct target compositions were utilised, along with varying deposition parameters. Long-term oxidation experiments were conducted in a conventional furnace at temperatures of 850 °C for durations of 24 h up to 500 h. After the oxidation processes, we conducted an analysis using X-ray diffraction (XRD), focused ion beam (FIB) techniques, and transmission electron microscopy (TEM). Additionally, we performed low-temperature hot corrosion (LTHC) experiments at 700 °C with a hot gas corrosion testing rig, varying the concentrations of SO_2.

In summary, the study demonstrates that $Ti_{1-x-y-z}Al_xTa_ySi_zN$ coatings exhibit extremely low oxidation kinetics, suggesting long-term stability well beyond 500 hours, alongside excellent resistance to hot gas corrosion environments. Based on these results, cathodic arc-evaporated Ti-Al-N-based coating materials are also promising candidates for protective applications in the aviation and power generation sectors.

[1] R. Hollerweger et al., Surf. Coat. Technol. 257 (2014) 78–86.
[2] C.M. Koller et al., Surf. Coat. Technol. 259 (2014) 599–607.
[3] Z.R. Liu et al., J. Alloys Compd. 917 (2022) 165483.
[4] A.R. Shugurov et al., Vacuum. 216 (2023) 112422.
[5] X. Sun et al., Surf. Coat. Technol. 461 (2023) 129428.

2:00pm **MA1-2-MoA-2 Fabrication, Characterisation and Tribological Testing of Magnetron Sputtered Cr Coated Zr Alloy Cladding for Enhanced Accident Tolerance in Light Water Reactors***, Thais Netto,* Manchester Metropolitan University, Brazil*; Adele Evans,* Manchester Metropolitan University, UK*; David Goddard, Jack Cooper,* United Kingdom National Nuclear Laboratory, UK*; **Peter Kelly [peter.kelly@mmu.ac.uk]**,* Manchester Metropolitan University, UK

Research into accident-tolerant fuels (ATFs) for light water reactors (LWRs) has focused on improving the safety of zirconium alloy fuel rod claddings and one of the more developed approaches is the use of chromium coatings deposited onto the claddings. In addition to performing in oxidising conditions, normal operation also causes fretting wear on the fuel rod surface, which requires tribological improvements.

The aim of this work, therefore, is to produce Cr coatings using the magnetron sputtering technique for Zr alloy nuclear fuel rod cladding material to enhance oxidation and mechanical resistance. The coatings were characterised, as a function of deposition conditions, in terms of their morphology, topography, hardness, reciprocating and fretting wear resistance, scratch test performance and oxidation resistance in autoclave and air oxidation tests.All the coatings provided excellent oxidation protection, in comparison to the uncoated samples. Mechanical testing indicated contrasting results with coatings with higher hardness showing enhanced wear protection, but lower coating hardnesses provided better scratch test performance and reduced fretting wear.

Scale up of these experiments has progressed from small flat coupons, through short (<20cm) rods, to full length (4m) fuel rods.

2:20pm **MA1-2-MoA-3 Second Phase-Driven Surface Engineering Strategies for Corrosion and Oxidation Protection of Mg–8Al–4Ca Alloy***, **Yueh-Lien Lee [yuehlien@ntu.edu.tw]**,* National Taiwan University, Taiwan **INVITED**

This study elucidates how second phases, particularly the β-Al-Ca intermetallic, influence the behavior and corrosion performance of the non-flammable Mg–8Al–4Ca alloy during cerium conversion coating and micro-arc oxidation (MAO) treatments. Scanning Kelvin probe force microscopy (SKPFM) and transmission electron microscopy (TEM) analyses reveal that the β phase exhibits a lower Volta potential and higher electrochemical activity than the α-Mg matrix, serving as a micro-galvanic anode that accelerates localized corrosion and hydrogen evolution. During the cerium conversion process, this activity disrupts film uniformity; however, a simple deionized-water pretreatment dissolves the exposed β phase into $Al(OH)_3$, promoting homogeneous Ce deposition with enhanced coating integrity and corrosion resistance. In MAO processing, the distribution and conductivity of β phases strongly affect discharge behavior and coating development. At low voltages, localized discharges near β regions lead to thinner and non-uniform films, while higher voltages facilitate the formation of Mg–Ca-rich silicate/oxide phases that improve corrosion resistance but hinder further thickening. Selective removal of surface β phases prior to MAO yields thicker and more uniform coatings. These findings clarify the mechanistic link between second-phase characteristics and coating evolution, providing effective strategies to engineer durable protection for non-flammable magnesium alloys.

3:00pm **MA1-2-MoA-5 Development of High-Temperature Ceramic Bond Coats for Environmental Barrier Coatings***, **Rebekah Webster [rebekah.webster@nasa.gov]**, Benjamin Kowalski, Bryan Harder,* NASA Glenn Research Center, USA

Environmental barrier coatings (EBCs) have enabled the use of silicon carbide (SiC)-based ceramic matrix composites (CMCs) in gas turbine engines by protecting the underlying CMC from corrosive combustion species such as water vapor. EBCs currently in service consist of a silicon bond coat and a rare earth silicate topcoat. The melting point of the silicon bond coat (~1410°C) limits the upper use temperature of these coating systems. To protect SiC-based CMCs at temperatures beyond that achievable by the current state-of-the-art, a mullite-based bond coat capable of withstanding temperatures of up to 1482°C has been developed at NASA Glenn Research Center. The mullite-based bond coat can be deposited by either plasma spray physical vapor deposition (PS-PVD) or slurry processing, and various EBC architectures with this bond coat have been developed and tested. In this work, the performance of these EBCs under oxidizing environments including steam and temperature cycling are reviewed, with the effect of coating microstructure, namely porosity, on oxidation being highlighted.

3:20pm **MA1-2-MoA-6 Statistical Correlation between Microstructural Features and Process Forces in Conventional and Ultrasonic-Assisted Milling of Plasma Claddings***, Kai Treutler,* TU Clausthal, Germany*; **Dirk Schröpfer [dirk.schroepfer@bam.de]**,* Bundesanstalt für Materialforschung und -prüfung, Germany*; Maraike Willeke,* TU Clausthal, Germany*; Thomas Kannengießer,* Bundesanstalt für Materialforschung und -prüfung, Germany*; Volker Wesling,* TU Clausthal, Germany

The development of highly efficient and economical steel components in plant and process engineering is crucial for reducing CO2 emissions. To withstand the high combined corrosive, tribological, thermal, and mechanical stresses, wear-resistant coatings tailored to the application and steel grade are employed. The increasing demand to substitute conventional cobalt alloys with nickel alloys, coupled with the need for defined or functional surfaces of high integrity, necessitates the development of novel wear-resistant coatings.

The use of wear-resistant coatings is essential for highly efficient and economical steel components in equipment, process, and power plant engineering. Co-alloys are commonly used as wear-resistant coatings for steel components, tailored to the specific application. The substitutability of Co alloys with Ni-based wear protection systems, in addition to price and supply uncertainties, is facilitated by the combination of innovative welding and machining processes such as ultrasonic-assisted milling.

This study investigates the influence of the microstructure and precipitation morphology adjusted by means of alloy modification on the machinability of wear-resistant plasma cladded coatings. The wear protection alloy NiCrMoSiFeB (trade name: Colmonoy 56 PTA), typically used for screw machines, is employed as a model system. Metallurgical investigations and in-situ measurements of occurring process forces and temperatures at the tool cutting edge during milling, as well as subsequent investigations of tool wear and surface integrity, allow for a detailed analysis and correlation between microstructural properties and machinability.

The primary objective of this study is the statistical correlation between specific microstructural features, like precipitation size, shape and amount with the characteristic process forces of conventional and ultrasonic assisted milling of the claddings.

The addition of Al, Ti, or Nb to the cast samples results in a clear change in the microstructure, hardness and machinability. Al and Ti cause long-needled or star-shaped precipitations and hardness increases, which lead to higher cutting forces and increased tool wear. In the case of the modified alloys, the inclusion of the alloying element Nb results in the formation of a more refined hard phase and reduces the machining force required for C56.

In most cases, the wear resistance potential has been maintained. The statistical model allows to adjust the chemical composition to a better machinability of the hard facings.

4:00pm **MA1-2-MoA-8 Oxidation Resistance of Binary and Ternary Nitrides Obtained by Magnetron Sputtering**, ***Ludovic Mereaux [ludovic.mereaux@unilim.fr]***, IRCER, France; *Edern Menou, Thomas Vaubois,* Safran, France; *Cédric Jaoul,* IRCER, France; *Marjorie Cavarroc,* Safran, France

Increasing aircraft engine temperature is one method, amongst others, to decarbonize aviation. But at high temperature, metallic materials performances are drastically decreased due to the effect of hot corrosion. To limit this impact, metallic materials need to be protected with dedicated coatings with adequate properties, which “entropy-augmented” ceramics could feature.

However, the composition space of complex ceramics is very wide, and comparatively very few bibliographical data are available as these specific ceramics have not been widely studied to date. While the use of data-driven approaches to identify relevant compositions appears necessary, it is not sufficient as (1) it requires data to be trained on, and (2) final properties should be experimentally assessed.

Nitride coatings obtained by PVD methods have been used in the machine tool and aerospace industries for many years. Binary nitride systems exhibit mechanical properties such as high hardness (20-25 GPa [1]). The addition of transition metals, such as Al or Si, improves physical and chemical properties like wear resistance, thermal stability and oxidation resistance [2].

Two main challenges have to be overcome: achieving a single solid solution film to guarantee both material and property homogeneity throughout the coatings, and assessing the long-term mechanical and environmental stability of the materials.

It was decided to creates our own database, starting from simple binary coating with the progressive addition of elements. In this talk, we will present results on the oxidation of binary and ternary nitride coatings. These coatings are obtained by magnetron sputtering in a reactive atmosphere and they are annealed in air up to 900°C, to propose oxidation mechanisms.

[1] W. D. Sproul, M. E. Graham, M.-S. Wong, et P. J. Rudnik, « Reactive unbalanced magnetron sputtering of the nitrides of Ti, Zr, Hf, Cr, Mo, Ti-Al, Ti-Zr and Ti-Al-V », *Surface and Coatings Technology*, vol. 61, nº 1, p. 139–143, déc. 1993, doi: 10.1016/0257-8972(93)90216-B.

[2] V. Novikov, N. Stepanov, S. Zherebtsov, et G. Salishchev, « Structure and Properties of High-Entropy Nitride Coatings », *Metals*, vol. 12, nº 5, p. 847, mai 2022, doi: 10.3390/met12050847.

4:20pm **MA1-2-MoA-9 Adaptive Opto-Neuromorphic Device Based on Monolayer MoS_2 for Extreme-Temperature Cognitive Operations**, ***Pukhraj Prajapat [pukhraj.npl@gmail.com]***, *Govind Gupta,* National Physical Laboratory, India

High-temperature neuromorphic devices are becoming increasingly essential as technology advances to support space exploration and withstand extreme conditions, such as those found in factories. To overcome this need, the researchers are devising technologies that imitate the human brain structure and operation. In this work, we present a scalable neuromorphic device based on a monolayer of MoS_2 that demonstrates operation at 100°C. The device portrays excellent electrical performances, mostly due to the great thermal stability of monolayer MoS_2 and its mechanical flexibility. Among these performances are low power consumption, fast switching, high resistance ratio, low switching voltage, and long stable endurance (~10^3 cycles). Besides, the device mimics neuromorphic behaviour by embedding the synaptic plasticity that is the major functional property of biological neural networks, thus allowing advanced cognitive computing in extreme environments. This is the first step toward a combination of materials science and neuromorphic computing, and it clears the way for smart resilient electronics that could survive in a variety of harsh conditions. This research aimsto achieve a significant breakthrough in the field of high-temperature electronics, paving the way for the development of future high-performance electronics that can meet the demands of modern technology.Keywords: 2D, TMDCs, Neuromorphic, Brain inspired, MoS2

4:40pm **MA1-2-MoA-10 Reactive Sputtering of CrMoNbWxTiCy Carbide Films by High Power Impulse Magnetron Sputtering System: Effect of W and Carbon Contents**, ***ChunHao Cheng [itsjonardgx@gmail.com]***, *Yung-Chin Yang,* National Taipei University of Technology, Taiwan; *Jyh-Wei Lee,* Ming Chi University of Technology, Taiwan; *Bih-Show Lou,* Chang Gung University, Taiwan; *Chia-Lin Li,* Ming Chi University of Technology, Taiwan

High power impulse magnetron sputtering (HiPIMS) has attracted significant attention for its ability to generate high-density plasma and achieve highly ionized metal species. Compared with conventional DC sputtering, this technique enhances ion bombardment energy, leading to improved film densification and adhesion. In this study, CrMoNbTiWx and CrMoNbWxTiCy high entropy alloy (HEA) coatings were deposited using HiPIMS to investigate the effects of tungsten and carbon incorporation on the microstructure and multifunctional properties of HEA carbide films. By varying the W target contents and controlling the reactive acetylene gas flow rates, the influence of target poisoning on film growth behavior was systematically analyzed. The film morphology and phase structure will be examined using field-emission scanning electron microscopy, X-ray diffraction, transmission electron microscopy, and atomic force microscopy. Mechanical properties, including hardness, adhesion, and wear resistance, will be characterized by nanoindentation, scratch, and pin-on-disk wear tests. Corrosion and oxidation resistance will be evaluated through a potentiodynamic polarization test in 3.5 wt.% NaCl solution and thermogravimetric analysis, while electrical performance will be assessed using a four-point probe to measure their electrical resistivities. This research aims to elucidate the roles of tungsten and carbon contents, as well as target poisoning behavior, in optimizing the structural, mechanical, and electrochemical performance of CrMoNbTiWxCy HEA carbide coatings, demonstrating the advantages of HiPIMS for developing dense and durable multifunctional protective films.

5:00pm **MA1-2-MoA-11 Materials for Aerospace Extreme Environments**, ***Samir M. Aouadi [samir.aouadi@unt.edu]***, University of North Texas, USA **INVITED**

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