Coatings for Use at High Temperatures
Room Pacific E - Session A1-1-MoM

Coatings to Resist High-temperature Oxidation, Corrosion, and Fouling I

Moderators: Dr. Sebastien Dryepondt, Oak Ridge National Laboratory, USA, **Gustavo García-Martín**, REP-Energy Solutions, Spain

10:00am **A1-1-MoM-1 Bill Sproul Award and Honorary ICMCTF Lecture: Strategies for the Development of Robust and Stable, but also Functional Ceramic Coatings*, Paul Mayrhofer*[1]**, TU Wien, Institute of Materials Science and Technology, Austria **INVITED**

For mechanically dominated load profiles, nitrides are preferred, while oxide materials offer better protection against high-temperature corrosion. Thus, when mechanical and thermal loads are combined, the nitrides used should also have excellent temperature and oxidation resistance. How to develop such nitride materials that can withstand both high mechanical and thermal loads will be the focus of this presentation. In addition, we will also discuss the excellent supercapacitor properties of transition metal nitrides.

Using transition metal nitride coatings, we will discuss important guidelines for material development to improve strength, fracture toughness, and stability. In particular, the stability (emphasis on phase stability to composition and temperature, but also to oxidation) of nitrides is a highly interesting task. For example, while the face-centered cubic (fcc) structure of TiN_x has a relatively large homogeneity range, the fcc structure of other transition metal nitrides (such as MoN_x and TaN_x) is extremely sensitive to small chemical variations, even if only the vacancy concentration changes. We will use these model systems to explore the possibilities of alloy and structural developments.

Among the many alloying elements that have been studied for (Ti,Al)N-based coatings, tantalum is one of the most versatile, capable of simultaneously increasing strength, fracture toughness, thermal stability, and oxidation resistance. This can be further improved when alloyed with Si and reactive elements.

The concept of high entropy is also very beneficial for hard ceramic thin films. We will see that, for example, (Hf,Ta,Ti,V,Zr)N and (Al,Cr,Nb,Ta,Ti)N easily outperform their commonly used binary or ternary constituents in terms of thermal stability and thermomechanical properties. In addition, all of the highly entropic ceramic sublattice thin films studied were relatively insensitive to variations in deposition parameters-which is good because their properties are at a high level.

With superlattice coatings, we will discuss how such nanolamellar microstructures can also simultaneously improve strength and fracture toughness.

However, we will also investigate the performance of electrochemical supercapacitors, which strongly depends on chemical stability, accessible surface area, and electrical conductivity. Transition metal nitrides are also excellent candidates for this purpose, but must have a very open-pore microstructure. Glancing angle deposition enables the fabrication of such zigzag-structured electrodes based on γ-Mo_2N, combining excellent electrochemical energy storage capabilities with excellent mechanical flexibility.

The individual concepts allow the materials to be designed to meet the ever-growing demand for further coatings tailored to specific applications.

10:40am **A1-1-MoM-3 Ti_5Si_3/$TiAl_3$ Multilayer Coatings as Oxidation Protection for γ–TiAl*, Peter-Philipp Bauer,*** German Aerospace Center and Brandenburg University of Technology Cottbus, Germany*; R. Swadźba,* Łukasiewicz Research Network - Institute for Ferrous Metallurgy, Poland*; L. Klamann,* German Aerospace Center, Germany

Titanium aluminides exhibit a high specific strength and a decent oxidation resistance up to 800 °C. This renders TiAl an excellent structural material for turbine blades. With the intention to increase the oxidation resistance at temperature above 800 °C, a large variety of different oxidation protection coatings were developed. It turned out that a combination of a diffusion inhibiting interlayer consisting of the Ti_5Si_3 phase and an oxidation resistant top layer of the $TiAl_3$ phase provided an excellent oxidation resistance. As a further development, a novel multilayer coating system of alternating Ti_5Si_3 and $TiAl_3$ layers were introduced. This design is expected to have exhibit a good oxidation resistance but also the high crack tolerance.

The coating system was produces by a continuous process using magnetron sputtering with elemental Ti, Al and Si targets. The performance of the coating was evaluated by cyclic oxidation tests in air at 900 °C for 1000 cycles (1h each) combined with thermogravimetric analysis. Scanning and transmission microscopy as well es x-ray diffraction was used to trace the oxidation and phase transformation processes.

Contrary to the expectations, the multilayer coating system showed only an insufficient oxidation protection. Although a good oxidation resistance was given during the first 100 cycles, at longer oxidation times the coating suffered under sever oxidation. In this talk, the failing mechanism as well as the lessons learned will be presented.

11:00am **A1-1-MoM-4 Max-Phase Based PVD Coatings as Protection for Lightweight Materials in High Temperature Environments*, Nadine Laska,*** *R. Anton,* German Aerospace Center, Germany*; R. Swadzba,* Lukasiewicz Research Network - Institute for Ferrous Metalurgy, Poland*; P. Nellessen,* German Aerospace Center, Germany

MAX-phases are of increasing interest as coating material for high temperature applications due to their unique combination of metallic and ceramic properties. Especially the alumina forming MAX phases of Cr_2AlC, Ti_2AlC or Ti_2AlN are promising as oxidation resistant coatings. Unfortunately, degradation of MAX phases is observed when applied on various Ti- or Ni-based alloys by interdiffusion processes between coating and alloy and the associated Al-depletion. This degradation is not present when MAX-phases are applied on the Al-rich γ-TiAl based alloys, which leads to an inward diffusion of Al from the substrate alloy into the coating and finally to a stabilization of the thermally grown alumina layer.

In the present work, the coating deposition process to get the MAX phases was DC magnetron sputtering using pure elemental targets of Ti or Cr, Al and C and in case of the Ti_2AlN MAX-phase based coating, nitrogen as reactive gas. No additional heating was applied during the sputtering process, the obtained substrate temperature was self-adjusted due to the target power. Prior to coating deposition, an Ar-plasma etching process for surface cleaning using a bias voltage of 500V and a frequency of 100 kHz was carried out for 15min. Using a threefold rotation, homogenous all-around coatings of about 10 µm were achieved with the desired stochiometric composition of the MAX-phases. The formation of the MAX-phase in the sputtered coatings was characterized during a post-annealing process at 800°C by in situ HT-XRD measurements as well as by SEM equipped with EDS and WDS, as well as by TEM with electron diffraction.

The MAX-phase coatings were tested under cyclic oxidation conditions. They provide a good oxidation protection of the γ-TiAl alloys due to the development of a protective alumina layer up to 850°C for up to 300 hrs in laboratory air. The performance of the MAX-phases is strongly depended on the substrate material and the accompanying interdiffusion processes between coating and substrate. Therefore, the Ti-Al-C based coating is more favored on TiAl alloys due to the thermodynamic stability of the Ti_2AlC MAX phase in particular in the presence of the γ-TiAl phase. In comparison, the Cr_2AlC MAX phase degrades after just 100 hrs at 850°C due to the formation of chromiumcarbides next to alumina.

11:20am **A1-1-MoM-5 Oxidation behaviors of (AlCrSiTi)N coatings on AISI 304 steel: A Combinatorial Study*, Sheng-Yu Hsu,*** *S. Chang, J. Duh,* National Tsing Hua University, Taiwan

Hard protective coatings have been widely applied in manufacturing industries to improve the performance and durability of workpiece. Scientists and engineers have dedicated to develop advanced coating materials which can be operated in harsh environments, e.g., electrochemical corrosion, high temperature oxidation. However, developing new materials has always been a crucial yet time-consuming task in materials science and engineering.

In this study, an experimental combinatorial approach via co-sputtering technique to efficiently investigate the effect of coating composition on the high temperature oxidation behaviors of (AlCrSiTi)N-coated AISI 304 steel under 700°C is demonstrated. From the elemental quantification results of 1-hour oxidized (AlCrSiTi)N, the oxygen content strongly correlates to the as-deposited coating composition, showing highest oxygen content (lowest oxidation resistance) of Ti rich composition and lowest oxygen content of Cr rich composition. TEM characterization exhibits that three oxide layers are formed after oxidation: spinel-Cr_2MnO_4 (originated from substrate diffusion), corundum-Cr_2O_3, and a thin layer of nano-crystalline mixed oxide. Except for Ti rich composition, an additional TiO_2 layer forms at the outermost layer. This study successfully demonstrates the efficiency and efficacy of developing advanced coating materials of superior high

[1] **Bill Sproul Awardee**

temperature oxidation resistance via experimental combinatorial approach.

11:40am **A1-1-MoM-6 Enhanced Pitting Resistance of Cathodic Arc Evaporated AlCrXN Coatings***, O. Hudak, F. Bohrn, P. Kutrowatz, T. Wojcik,* Christian Doppler Laboratory for Surface Engineering of high-performance Components, TU Wien, Austria*; E. Ntemou,* Ion Physics Group, Department of Physics and Astronomy, Uppsala University, Sweden*; D. Primetzhofer,* Ion Physics Group, Department of Physics and Astronomy, Uppsala University, Austria*; L. Shang, O. Hunold,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein*; P. Polcik,* Plansee Composite Materials GmbH, Germany*; **Helmut Riedl**,* Institute of Materials Science and Technology, TU Wien, Austria

With current state-of-the-art corrosion resistant coatings being far from optimized, it is of great interest to further investigate the fundamental mechanisms and underlying driving forces that dominate the degradative process.Particularly saline environments represent a technological frontier, where high-performance components suffer accelerated breakdown through a localized corrosion mechanism called-- pitting. Porosities, macroparticles, and the overall columnar growth morphology of physical vapor deposited coatings makes them particularly susceptible to inward-diffusion of corrosive media. Here, especially chloride ions play a key-role allowing for an accelerated attack at the coating substrate interface.

In a first step, this study provides a systematic approach on highlighting preferred diffusion pathways of corrosive NaCl-rich media of $Al_{0.7}Cr_{0.3}N$-based PVD thin films deposited on low alloy steel. Through an array of high-resolution techniques, such as TEM, ToF-SIMS, APT and t-EBSD, we intend to break down the possible diffusion paths from a micrometer to a nanometer scale, providing newest insights on the corrosion process. In a second step, this study showcases a doping strategy, as well as thermal treatment as viable approaches for improving the corrosion behavior of the previously discussed AlCrN system. In order to investigate the beneficial effects of the dopant, a series of $Al_{0.7}Cr_{0.3-y}X_yN$ coatings were deposited with varying alloying contents. Electrochemical tests of the as-deposited, as well as thermally treaded coatings were conducted using a three-electrode cell set up, whereupon extrapolations of Tafel-plots were used to evaluate the corrosion resistance.

Keywords: Corrosion Resistance; Pitting; Cathodic Arc Evaporation; PVD coatings; Diffusion Pathways;

12:00pm **A1-1-MoM-7 Novel Approaches for the PVD Synthesis of Advanced Aluminide Thin Films: The Example of Ruthenium-Aluminide***, **Vincent Ott**,* Karlsruhe Institute of Technology (KIT), Institute for Applied Materials (IAM), Germany*; T. Wojcik,* TU Wien, Austria*; S. Ulrich,* Karlsruhe Institute of Technology (KIT), Institute for Applied Materials (IAM), Germany*; S. Kolozsvári, P. Polcik,* Plansee Composite Materials GmbH, Germany*; P. Mayrhofer, H. Riedl,* TU Wien, Austria*; M. Stueber,* Karlsruhe Institute of Technology (KIT), Institute for Applied Materials (IAM), Germany

Transition metal aluminides are known for their use as high temperature materials in aerospace and gas turbine engine applications. Their best-known representatives of these intermetallics are NiAl, TiAl and FeAl. All these materials suffer from brittle behavior at room temperature and limited operating temperature of approx. 800°C. A relatively new and unknown candidate with improved properties regarding ductility, toughness and high temperature resistance is the RuAl. Thin film synthesis can enable the exploitation of their full potential for example as protective coatings at high temperature conditions. To elucidate this potential, RuAl single layer thin films were synthesized by magnetron sputtering.Different approaches were conducted, including a multilayer thin film approach combined with a post-annealing as well as direct magnetron sputtering from a compound target to study the formation of the cubic B2 RuAl phase. Depending on the synthesis route, different microstructures and corresponding properties of the thin films were obtained, analyzed by electron microscopy and XRD techniques.

Coatings for Use at High Temperatures
Room Pacific E - Session A1-2-MoA

Coatings to Resist High-temperature Oxidation, Corrosion, and Fouling II

Moderators: Gustavo García-Martín, REP-Energy Solutions, Spain, **Dr. Justyna Kulczyk-Malecka**, Manchester Metropolitan University, UK

1:40pm **A1-2-MoA-1 Microstructural Changes of Yttria-Containing MMC-Coatings and Their Influence on Hot Corrosion, Wear and Mechanical Behavior***, **Christoph Grimme**, C. Oskay, M. Galetz,* DECHEMA-Forschungsinstitut, Germany

While very high inlet temperatures increase the efficiency of turbines, they lead to the requirement of oxidation resistant bond coats for TBC systems. MCrAlY (M: Ni and/or Co) coatings are used for turbine blades in a broad field of applications. However, their deposition processes by HVOF or EB-PVD are cost-intensive and limited by line-of-sight. Galvanic co-deposition methods can be utilized to apply these types of coatings at lower cost and without line-of-sight limitations. This technique also opens the possibility to incorporate different metallic and ceramic particles alongside with galvanically deposited Ni, Co, and Ni/Co. Coatings for turbine components should not only improve the oxidation resistance of the system, but also increase the hot corrosion resistance to ensure an extended lifetime. One of the most aggressive attack in turbines originates from vanadium present in the fuel to be burned in gas turbines. It forms eutectic ashes with Na_2SO_4 from fuel or air ingestion of NaCl to form low melting V_2O_5/Na_2SO_4 compounds [1].

In this study, a novel galvanic co-deposition method was used to incorporate yttria particles in the Ni-coatings. Thereafter, the pristine Ni/Y_2O_3 coatings were enriched with Al and/or Cr by pack cementation. By the co-depositon of yttria alongside with nickel not only the oxide formation, but also the oxide adherence could be strongly improved due to the reactive element effect [2]. It was proven, that Y_2O_3 is also able to react with V_2O_5 to form high melting YVO_4 and thereby avoiding the formation of highly corrosive, low melting V_2O_5/Na_2SO_4 compounds. The influence of Y_2O_3 on oxidation and hot corrosion, as well as wear and mechanical properties compared to unmodified NiAl coatings was studied.

It was found that additions of nanosized Yttria in the Ni-coating cause a significant grain refinement after the aluminum diffusion process, with the grains being much larger for yttria-free coatings. Hardness measurements revealed an increase of approx. 100 HV1 for MMC NiAlY coatings compared to coatings without dispersed nano–Y_2O_3 particles. Furthermore, significant improvements of the thermocyclic oxidation behavior and hot corrosion resistance were achieved by the incorporation of Y_2O_3 to the metallic coatings.

[1]X. Montero, M. C. Galetz,*Oxid. Met.***2018**, *89*, 499–516.

[2]J. Stringer,*Mater. Sci. Eng. A***1989**, *120*, 129–137.

2:00pm **A1-2-MoA-2 Surface Refinement by Aluminide Diffusion Coatings and Its Effect on the Oxidation Behavior and Creep Strength of Additively Manufactured Fe- and Ni-Based Alloys***, **Ceyhun Oskay**, L. Mengis,* DECHEMA-Forschungsinstitut, Germany*; A. Kulig, H. Daoud,* Neue Materialien Bayreuth GmbH, Germany*; M. Galetz,* DECHEMA-Forschungsinstitut, Germany*; U. Glatzel,* University of Bayreuth, Germany and Neue Materialien Bayreuth GmbH, Germany

Additive manufacturing (AM) has created new possibilities for the rapid production of metallic components and has received considerable interest by a variety of industries such as chemical, energy and transportation, since this form of production of parts and/or spare parts can lead to the independence from supply chains. Furthermore, AM can offer the tailoring the component design, microstructure, and mechanical properties according to the requirements [1]. One of the major drawbacks of the AM is the requirement of a post-processing, since the components in the as-printed condition always possess a significantly high surface roughness and high internal stresses. This leads to a deterioration of their service-relevant properties such as oxidation resistance and fatigue strength. The industrial surface post-processing is usually conducted by grit blasting and heat treatment and is therefore usually difficult to apply for large parts with complex geometries. Even after these post-processing, AM components may still require improvement in their oxidation resistance, as process temperatures steadily increase for higher efficiencies. At higher service temperatures, the enrichment of Al at the surface by diffusion coatings has been shown to be very effective to protect Fe- and Ni-based alloys from oxidation. Such coatings form and maintain a slow-growing and dense alumina scale [2,3].

In this study, pack cementation was utilized to deposit aluminide diffusion coatings on additively manufactured (via Laser Powder Bed Fusion, LPBF) Fe- and Ni-based alloys to chemically modify and refine their surface simultaneously. A significant reduction of the surface roughness as well as sealing of the pores in the vicinity of the surface was observed after the coating deposition. Quasi-isothermal oxidation tests up to 1000 h were conducted at 800°C and 1000°C in laboratory air for aluminized and uncoated LPBF specimens. A significant improvement of the oxidation behavior was observed for the aluminized LPBF alloys compared to uncoated alloys. Creep tests were conducted at 800°C and 1000°C to characterize the performance of LPBF alloys with and without aluminide coatings. These results were compared with aluminized and uncoated wrought samples under the same conditions. Furthermore, load effects on scale formation and re-healing, depletion of the oxide former and crack formation were discussed for the AM alloys.

[1] I.E. Anderson et al., Curr. Opin. Solid State Mater. Sci. 22 (2018) 8-15.

[2] D.K. Das, Prog. Mater. Sci 58 (2013) 151-182

[3] B.L. Bates et al., Surf. Coat. Technol. 204 (2009) 766-770.

2:20pm **A1-2-MoA-3 Influence of High Temperatures on the Friction and Wear of Highly Stressed Exhaust Systems***, **Martin Dienwiebel**,* Institute for Applied Materials IAM - Karlsruhe Institute of Technology, Germany*; T. König,* Fraunhofer Institute for Mechanics of Materials IWM, Germany*; T. Kimpel,* Institute for Applied Materials IAM, Karlsruhe Institute of Technology, Germany*; D. Kuerten, A. Kailer,* Fraunhofer Institute for Mechanics of Materials IWM, Germany **INVITED**

We investigated the atmospheric effect of exhaust components on the wear of cast iron against chromium plated steel at temperatures up to 800 °C. Reciprocating wear tests of a cylinder plate configuration were performed in air and a low-oxygen CO_2-N_2-O_2 atmosphere and analyzed afterwards.

At temperatures above 400 °C a tribological induced oxide layer is formed at the interface, a so-called “glaze layer”, which replaces an adhesive regime and leads to a strong decrease of wear. The investigation proves a layered structure out of a porous lower and a highly compacted upper part with different chemical compositions. A change of atmospheres shows low impact on this tribological mechanism above a threshold temperature of 400 °C, assuming sufficient oxidation times of the generated wear particles, which agglomerate, were compacted and sintered due to the tribological stresses and temperatures. Based on this finding, a temperature related sinter or phase transition process is postulated to determine the glaze layer formation independently of comparable small atmospheric differences. At the adhesion dominated regime of lower temperatures a Carbon enriched layer of 400 nm thickness was observed in the CO_2-N_2-O_2 atmosphere and is made responsible for a decrease of wear.

3:00pm **A1-2-MoA-5 Surface Refinement of Additively Manufactured Components: Microstructure and Mechanical Properties***, **Agata Kulig**,* Neue Materialien Bayreuth GmbH, Germany*; C. Oskay, L. Mengis,* DECHEMA-Forschungsinstitut, Germany*; H. Daoud,* Neue Materialien Bayreuth GmbH, Germany*; M. Galetz,* DECHEMA-Forschungsinstitut, Germany*; U. Glatzel,* University of Bayreuth, Neue Materialien Bayreuth GmbH, Germany

Laser Powder Bed Fusion (L-PBF) is already used for manufacturing complex parts with high precision. However, the high cost of surface finishing is still a limiting factor for the spread of L-PBF components in various industries. The high surface roughness and their resulting residual porosity influence the mechanical properties, especially dynamic ones. In this presentation, the pack cementation process will be introduced as a novel method to refine the surface roughness and the microstructure in the surface zone as well as to improve the mechanical behavior of L-PBF parts.

The effect of the pack cementation process on the resulting microstructure in the surface zone of L-PBF specimens was investigated. For this purpose, different Fe- and Ni-Basis alloys were investigated. The surface roughness of as-built and as-modified L-PBF specimens was measured. Finally, the tensile strength as well as the fatigue behavior of both as-built and as-modified specimens will be presented.

3:20pm **A1-2-MoA-6 Oxidation Behavior of Novel Cr-Si Diffusion Coatings Applied by the Slurry Technique***, **Michael Kerbstadt**,* DECHEMA, Germany*; E. White,* DECHEMA, USA*; M. Galetz,* DECHEMA, Germany

Diffusion coatings are widely used in high temperature applications to enhance the oxidation and corrosion resistance of metals and alloys. Commonly Al, Cr, or Si are enriched at the surface in order to form protective oxide scales during exposures at high temperatures. Al, Cr and Si-based diffusion coatings are mostly achieved by pack cementation, where the deposition occurs via a gas diffusion process. For pack cementation the substrates are usually fully embedded into a powder mixture, which is labor-intensive and requires extensive furnace fixturing to be heated-up during each run. For an Al diffusion coating, an alternative slurry process is well established, where the slurry is sprayed onto the metallic surfaces by an air brush. The coating is then established during a subsequent heat treatment by interdiffusion between the metallic particles from the slurry and the substrate. For sufficient diffusion rates during the heat treatment, the existence of a liquid phase at the interface of the substrate surface and the slurry particles is necessary. Because of the high melting point of Cr, the deposition of Cr-based diffusion coatings by the slurry technique has been challenging. Cr-Si slurry coatings have recently been successfully developed at the Dechema-Forschungsinstitut. These newly developed slurry coatings are applied using an aqueous suspension where distilled water is used as the solvent. Higher Cr-activities, due to the partial liquid state, enable higher coating thicknesses when compared to similar coatings applied by pack cementation. This creates a larger reservoir of the protective oxide-forming elements Cr and Si, resulting in longer-lasting protection from the coating.

In this work the oxidation behavior of novel Cr-Si slurry coatings applied on the austenitic steel Sanicro 25 and the Ni-base alloy Inconel 617 are investigated. A Cr and Si enriched diffusion zone of more than 100 µm could be achieved on both materials. Isothermal and cyclic (1h/cycle) oxidation exposures at 900°C for 1000 h in lab air were carried out. To classify the results, samples with coatings applied by pack cementation and also bare substrate materials were exposed under the same conditions. To determine the weight gain samples were removed after 300 h, 700 h and 1000 h. Analysis by XRD, SEM and EPMA was performed to determine the structure and composition of the oxide layers and the degree of oxidative attack. Due to the increased amount of protective elements, the slurry coated samples showed a lower overall mass gain and decreased oxidation attack during the exposures.

3:40pm **A1-2-MoA-7 Use of Machine Learning Algorithms to Optimize and Customize Aluminide Diffusion Coatings***, **Vladislav Kolarik**, M. Juez Lorenzo,* Fraunhofer Institute for Chemical Technology ICT, Germany*; P. Praks,* IT4Innovations National Computing Center, VSB - Technical University of Ostrava, Czechia

Aluminide diffusion coatings are a highly efficient and economic technique to protect steels against corrosion at high temperatures in aggressive media such as molten salts or steam. They are easy to apply using different methods of deposition such as spraying or brushing with a subsequent heat treatment to form the diffusion coating. Machine learning algorithms offer a huge potential for optimization as well as for customization of the coatings to a particular application with different substrate steels and media. The experimental effort can be minimized reducing the costs significantly and accelerating the development. In this context the present work investigates the use of machine learning to determine the coating process parameters that lead to the targeted coating characteristics and properties.

First, the entire coating system and its manufacturing process was fully parametrized considering every single parameter having influence on the coating. Variable input parameters have been defined: green slurry thickness, slurry viscosity, aluminum particle size, curing temperature and time, heat treatment temperature and time. Other input parameters such as the substrate steel, surface roughness, slurry composition, purity of the aluminum particles, slurry deposition method and air as atmosphere were kept constant. Output parameters characterizing the diffusion coating were defined: coating thickness, number of layers and their thicknesses, pores concentrations and FeAl precipitations in the Fe2Al5 layer. Assessment criteria for the targeted properties were defined, such as an overall thickness of 100 to 120µm, three allowed layers (Fe2Al5, FeAl, Fe+FeAl), and a porosity less than 5%. Algorithms from Machine learning such as CatBoost were chosen to undertake the first approaches. Experimental values from former projects were used to train the software for calculating the impact of process parameter variation on the coating properties and to validate the outcome.

4:00pm **A1-2-MoA-8 Self-Healing Aluminide Coatings***, **Fernando Pedraza**, R. Troncy, L. Boccaccini, G. Bonnet,* La Rochelle University, France*; X. Montero,* MTU, Germany*; M. Galetz,* DECHEMA-Forschungsinstitut, Germany

Upon high temperature exposure, the Al reservoir of the aluminium diffusion coatings is lost by oxidation (2-8%) and by interdiffusion (20-35%) [**1**]. This lowers oxidation and mechanical resistance in nickel-based superalloys. Thermodynamically stable γ/γ' $(NiPt)_3Al$-based coatings were thus considered [**2**] but they require appropriate contents of Pt and of Hf to provide adequate oxidation resistance [e.g. **3**]. Since the cost of Pt (and of Hf) is quite high, such stable coatings cannot be applied to the low pressure turbine components that operate at lower temperatures (T<1050°C) where simple aluminide coatings suffice [**4**]. The use of diffusion barriers has been proposed to arrest interdiffusion [e.g. **5**] but such layers can embrittle the whole coating system. In contrast, composite $NiAl$-Al_2O_3 coatings [**7**] display adequate mechanical properties [**6**] and the high exothermicity between preoxidized Ni powders or coatings and Al [**8,9**] is buffered, hence avoiding projection of molten metal [**10**].

Based on this, this paper investigates the synthesis of self-healing aluminide coatings made of a conventional nickel aluminide matrix where preoxidized Al-rich Ni_xAl_y microreservoirs are embedded [**11**]. The idea is that the oxide shell covering the microreservoirs governs the outward diffusion of the Al-rich intermetallic core whenever the matrix is depleted in Al upon oxidation. For such purpose, Ni_2Al_3 powders were first fabricated by pack aluminizing a Ni plate that was subsequently crushed and milled, preoxidized and then added a nickel-electroplating bath. A final slurry aluminizing process was conducted. The study was conducted on pure Ni as a model of the Ni-based superalloys. Subsequent isothermal oxidation at 1000°C was conducted for 48h in air. The main results show that the Al depletion in conventional simple aluminide coatings ranged between 32 and 45 at% while the ones containing the microreservoirs only lost 11 at% thereby showing the promising character of these coatings. The mechanisms of formation and of degradation of the coatings will be highlighted.

Refs

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4:20pm **A1-2-MoA-9 Continuous Al-supply to Cr_2AlC MAX Phase Coatings During Oxidation at High Temperature***, **Clio Azina**, M. Hans,* Materials Chemistry, RWTH Aachen University, Germany*; J. Gonzalez-Julian,* Chair of Ceramics, RWTH Aachen University, Germany*; P. Eklund,* Linköping University, IFM, Sweden*; J. Schneider,* Materials Chemistry, RWTH Aachen University, Germany

Phase stability is likely to be one of the most important specifications which determine the lifetime of materials operating in extreme environments. In the case of MAX phases, the weakly bonded A-elements diffuse along the basal planes when a thermal load is applied or when in presence of oxidizing environments. The A element then reacts with the oxidizing environment and forms a protective oxide scale. That is the case of the Cr_2AlC MAX phase in which Al diffuses to the surface and forms an Al_2O_3 scale. However, the Al depletion below the surface causes the local decomposition of the MAX phase into the binary carbide, Cr_7C_3.

In this work, the possibility of continuously supplying Al to Cr2AlC coatings is investigated in order to avoid the formation of the carbide layer. To this end, Cr2AlC substrates with different microstructures were used as substrates. These substrates were then coated with Cr2AlC by magnetron sputtering. The MAXonMAX assemblies were then oxidized in air, at high temperatures and the integrity of the assembly was assessed. Imaging allowed determining whether the different substrates allowed for Al transport across the substrate/coating interface and whether the concept of Al-supply from a MAX phase substrate to a MAX phase coating was viable. It appeared that the substrates played a major role in the oxidation behavior of the coatings as the oxide scale growth was impacted. This was attributed to the microstructure of the substrates which allowed for more or fewer diffusion channels depending on the grain size. Overall, the concept of Al-supply was shown to be successful when using fine-grained substrates but did not yield significant improvement when considering coatings grown on coarse-grained MAX phase substrate compared to those grown on conventional MgO substrates where Al supply does not occur.

Coatings for Use at High Temperatures
Room Pacific E - Session A1-3-TuM

Coatings to Resist High-temperature Oxidation, Corrosion, and Fouling III

Moderators: Gustavo García-Martín, REP-Energy Solutions, Spain, **Dr. Justyna Kulczyk-Malecka**, Manchester Metropolitan University, UK

8:00am **A1-3-TuM-1 Excellent Tribological, Mechanical, and Anti-Corrosion Performance of Agro-Waste as Corrosion Inhibitor for Carbon Steelin an Acidic Environment***, **Omotayo Sanni**, J. Ren, T. Jen,* Department of Mechanical Engineering Science, University of Johannesburg, South Africa

Presently, in the field of corrosion, the main goal is to develop environment-friendly and effective corrosion inhibitors that can be utilized to substitute conventional corrosion inhibitors, which are toxic. Therefore, the present work aims to investigate the microstructures, mechanical, corrosion, and tribological performances of agro-waste as a potential inhibitor for mild steel in 2 M HCl solutions by electrochemical impedance spectroscopy, weight loss, and polarization methods. Results obtained showed that the highest inhibition efficacy of 97.8% was obtained with a 500 ppm concentration of the waste. The potentiodynamic polarization test shows that the waste behaves as a mixed inhibitor. The electrochemical result shows that the charge-transfer resistance was increased, while, current density decreased with inhibitor in the 2 M HCl solutions. The inhibition performance of the waste increased with increasing concentration at the studied inhibitor dosages, the inhibitor molecules' adsorption on the metal fits the Langmuir adsorption isotherms. The experimental results showed that the waste products significantly improved resistance to wear. In addition, excellent toughness, high hardness, preferable adhesion, and good corrosion resistance also contributed to improve the tribological properties. The scanning electron microscope equipped with energy dispersive X-ray spectroscopy test confirms the protection of the mild steel in the HCl solutions. The results derived in this paper could prove highly beneficial and provide fundamental insights about the efficient use of agro-waste as an effective inhibitor, as well as stimulate the industrial application of agro-waste on a large scale.

8:20am **A1-3-TuM-2 Study of Materials and Coatings for Use in High Temperature CO_2 Environments***, **Jianliang Lin**,* Southwest Research Institute, USA

Oxy-fuel sCO2 power cycles are a transformational technology for the energy industry, providing higher efficiency heat source energy conversion for conventional and alternative energy sources. However, the technology requires advanced thermal management/protection systems to accommodate high temperatures for turbine critical components, e.g. nozzles and blades. The paper presents a study of NiCr based alloys and protective coatings for use in CO_2 environments using a high temperature thermal cyclic test rig. The thermal cyclic test was performed on uncoated and coated coupon samples in ambient atmosphere and CO_2 environment at 800 °C and 1150 °C, respectively. One testing cycle includes 50 min annealing at peak temperature and 10 min cooling in air. The tested NiCr based alloys include Haynes 230, 625 alloy, HR-120 alloy, Inconel 718, and C22 alloy. The coatings include a nanocrystlline MCrAlY coating with a TiN diffusion barrier which was deposited using plasma enhanced magnetron sputtering (PEMS) technique, and a thermal barrier coating (TBC) system (MCrAlY bond coat and YSZ top coat) deposited by air plasma spray (APS).The nanocrystlline MCrAlY coating and TBC aimed at providing thermal and oxidation protection for different turbine critical components that see low temperature (e.g. 800 °C) and high temperature (e.g. 1150 °C), respectively. During the thermal cyclic tests, accumulative mass changes of the test coupons were recorded. The microstructure, chemistry, and phase changes of alloys and coatings after thermal cyclic tests were characterized by scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD). The results showed that all uncoated alloys exhibited severe oxidation and degradation at 800 °C. The nanocrystlline MCrAlY coating alone provided sufficient thermal/oxidation protection for all alloys from 800 °C to 1050 °C in ambient atmosphere and CO_2 environment. However, additional TBC is needed for protecting the alloys for higher temperatures (>1150 °C).

8:40am **A1-3-TuM-3 Liquid Aluminum-Induced Wear of Ni-Based Superalloy at Elevated Temperatures***, **Hongfei Liu**,* Institute of Materials Research and Engineering (IMRE), A*STAR (Agency for Science, Technology and Research), Singapore*; N. Gong,* Institute of Materials Research and Engineering (IMRE), Singapore*; R. Karyappa, T. Meng,* Institute of Materials Science and Engineering (IMRE), Singapore

Ni-based superalloys have long been developed for structural components towards high-temperature applications, e.g., turbine blades, incineration plants, nuclear reaction plants, etc., due to their high mechanical strength and stable chemistry. On the other hand, along with the rapid development in additive manufacturing (AM) and remanufacturing of metal alloys in the past decades, electrochemical corrosion and hot corrosion have been extensively applied for testing and evaluating the chemical stabilities of AM-superalloys, in comparison with their competitors fabricated by traditional methods, at room and elevated temperatures.

Liquid metal has been employed in heat exchanger at high temperatures due to its higher thermal compacity than other media, e.g., water. In comparison to high-temperature oxidation and hot corrosion studies, liquid metal-induced wear of Ni-based superalloy is relatively less in the literature.

We have recently studied liquid aluminum-induced wear of Ni-based single crystal superalloys, addressed atomic diffusions and surficial cracks after cooling down from high temperatures. In this presentation, we will be discussing the experimental results and their related mechanisms.

9:00am **A1-3-TuM-4 Characteristics and Resistance of CVD Hafnium Carbide Coating in Extreme Environments***, **Hyeon-Geun Lee**, J. Lee, D. Kim, B. Jun, W. Kim, J. Park,* Korea Atomic Energy Research Institute, Republic of Korea

Hafnium carbide (HfC) has a high melting point and excellent resistance to ablation, corrosion, and mechanical/thermal stress. HfC coating is mainly considered to ultra-high temperature protective coating for aerospace application. HfC coating has strong anti-ablation ability due to high melting point, outstanding mechanical properties, superior oxidation resistance, absence of phase change at high temperature. Also, its favorable performance makes it possible to expect resistance in other extreme environments. Recently, in the field of nuclear power research, research on accident tolerant fuel (ATF) system and new generation power plant system including molten salt reactor (MSR) is in the spotlight. Structure materials are exposed to harsh environments such as hydrothermal corrosion and high temperature steam oxidation in ATF system and high temperature molten salt corrosion in MSR system. Various corrosion and oxidation resistance coatings including metal, ceramics, and hybrid coating that can protect structural materials in these extreme environments have been studied. In this study, characteristics and resistance of CVD hafnium carbide coating in various corrosion and oxidation environments were studied. Chemical vapor deposition (CVD) can provide the high purity dense coating with excellent crystallinity and uniformity. A highly crystalline dense HfC which contains a small amount excess carbon is uniformly deposited on graphite substrate using low pressure CVD from $HfCl_4$-C_3H_6-H_2 system. High temperature oxidation and ablation properties of CVD HfC coating were investigated. Hydrothermal corrosion evaluation is carried out using autoclave at 360 °C, 19 MPa condition. The steam oxidation resistance was investigated at up to 1600 °C temperature with maximum 200 cm/s flow steam condition. Corrosion and oxidation resistance of HfC were analyzed compared to SiC, which is known to be excellent. High temperature molten chloride salt corrosion experiment was conducted at 650 °C in the controlled oxygen and moisture environments. The possibility of CVD HfC coating as a corrosion resistant coating of metal structural material were researched.

9:20am **A1-3-TuM-5 High Temperature Corrosion Protection of Zirconium Fuel Rods in Nuclear Reactors by Nanocrystalline Diamond (Ncd) Layers***, **Frantisek Fendrych**,* Institute of Physics Academy of Sciences of the Czech Republic

Motivation

Nanocrystaline diamond (NCD) film can be utilized as a protective coating for zirconium alloy (Zircaloy2) nuclear fuel cladding (NFC) of nuclear reactors. One big disadvantage of Zircaloy2 is that it reacts with water steam and during this (oxidative) reaction releases hydrogen gas, which partly diffuses into the alloy forming zirconium hydrides. Moreover, the large production of hydrogen gas can result into catastrophic hydrogen-air explosions (as occurred in the recent Fukushima accident, March 11, 2011).

Plasma CVD reactor

Diffuse plasma in the linear antennas microwave plasma enhanced CVD reactor was used for coating of cylindrical Zircaloy2 rods with NCD films. The combination of the linear antennas arrangement and the use of low pressures, > 1 mbar, a diffuse large area plasma is formed enabling large area 3D NCD deposition.

Testing of the protective NCD films

We have successfully demonstrated the possibility to cover a cylindrical rod-shaped Zircaloy2 nuclear fuel cladding by a 300 nm thick protective NCD layer using the linear antennas microwave plasma enhanced CVD. NCD coated Zircaloy2 rods underwent a set of corrosion tests, namely a reactor irradiation test and hot steam oxidation. SEM, Raman, XRD, XPS were employed. Oxidation of NCD coated and uncoated ZIRLO at 1000 °C is presented in supplemental document on **Figs.1,2**. It confirms that a thin NCD layer can serve as a anticorrosion protective coating on NFCs in the harsh environment of a nuclear reactor at substantially elevated temperatures.

Conclusions

Zirconium alloy ZIRLO of nuclear fuel cladding was covered by 300 nm thick protective nanocrystalline diamond NCD layer using special Linear Antennas pulsed MicroWave Plasma Enhanced CVD deposition technique. The NCD layer protects ZIRLO rods surface against hydrogen penetration and against oxidation during standard reactor run at about300 °C hot water steam and significantly decreases both in accidental case of overheating up to 800 – 1100 °C. NCD anticorrosion protection can up to 40% prolong lifetime of ZIRLO fuel rods and consequently enhances the uranium dioxide UO2 nuclear fuel burnup what leads to more efficient use of nuclear fuel in reactors. Final NCD/ZIRLO samples are longtime (for 2 years) tested by Westinghouse for real permanent run in Halden Norway experimental nuclear reactor. Application of presented research results for anticorrosion protection of power stations nuclear reactorsis proposed in EU and USA patents **[1,2]**.

References

[1] EU Patent No. 3047046 / 2020, WO 2015039636, A1, 2015, PCT/2014/000101.

[2] USA Patent No. 10916352 / 2021, WRB-IP, Ref. 000036-016; U.S. App. 15/022,536, 2021.

9:40am **A1-3-TuM-6 Effect of Vacuum Annealing on the Residual Stress of ZrN Thin Film deposited on Ni-based Superalloy Haynes 282**, ***Kuan-Che Lan****, C. Li,* National Tsing Hua University, Taiwan*; H. Tung,* Institute of Nuclear Energy Research, Taiwan

Transition metal nitride thin films deposited using physical vapor deposition (PVD) methods tend to experience a significant amount of residual stress. The existence of residual stresses in thin films can do influence on their mechanical properties greatly. It is believed that the processing of heat treatment can help relief the residual stress in a crystalline material. To study the effect of heat treatment after a PVD method, ZrN thin films deposited using the DC magnetron sputtering system on both Si and nickel-based superalloy Haynes 282 substrates was annealed in vacuum (4×10^{-6} Torr) at 950˚C. After vacuum annealing, the film thickness, the crystallographic structure and depth compositional distribution of the annealed ZrN thin film will be characterized by scanning electron microscope, the X-ray diffraction and Auger electron spectroscopy, respectively. Averaged X-ray strain method will be also applied to analyze the residual stress of the ZrN thin film on Haynes 282.

10:00am **A1-3-TuM-7 Study at Pilot Plant Scale on Biomass Corrosion Resistance of FeCr and CoCrMo Coatings Applied by HVOF***, M. de Miguel Gamo, G. García Martín, M. Lasanta Carrasco, M. Lambrecht,* Universidad Complutense de Madrid, Spain*; F. Gonçalves,* Teandm - tecnologia engenharia e materiais s.a, Portugal*; M. Sousa,* teandm - tecnologia engenharia e materiais s.a, Portugal*; A. Bahillo, M. Benito,* Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas (CIEMAT), Spain*;* ***Francisco Javier Pérez Trujillo****,* Universidad Complutense de Madrid, Spain

The replacement of coal by biomass combustion is considered as a promising option to reduce the emissions associated to this fossil energy source. At the same time, the energetic use of the biomass contributes to solve the problem of waste disposal. On the other hand, this renewable technology presents two main disadvantages: (1) the cost of biomass is higher than coal, what leads to a higher cost of the electricity production, (2) biomass combustion environment is extremely corrosive induced by the high amount of chlorine present in the fuel (gas) and/or the present of alkali and heavy metals in condensed deposits. The aggressive conditions commonly imply the use of austenitic or nickel-based alloys that increases the investment required. An alternative to mitigate corrosion and allow operation at more aggressive conditions, is the use of highly-corrosion resistant coatings over lower cost alloys, such as ferritic steels.

The present study compares the biomass corrosion behavior of two different coatings, FeCr and CoCrMo, applied by High Velocity Oxy-fuel (HVOF) on a 12%Cr steel to improve its oxidation resistance. The coated specimens were tested in a pilot plant installation and exposed to the flue gases from the combustion of two different agricultural biomasses (eucalyptus and wheat straw) for 2000 h at 600°C. The samples were weight monitoring at different times during the testsand characterized before and after oxidation tests by X-ray diffraction (XRD) and scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS). At the end of the exposures, both coatings, FeCr and CoCrMo, have shown a significant corrosion layer. Although, when the results were compared with the 12%Cr steel uncoated, it was confirmed that the application of both coatings reduces the weight gain of the steel, increasing its corrosion resistance. The obtained results make these coatings a promising alternative, that should be further investigated from the mechanical perspective, for enhancing the lifetime of in-service power plant components.

10:20am **A1-3-TuM-8 Sol-Gel Coating to Protect Materials Exposed Under Carbonates Used as a Thermal Energy Storage System in Central Tower Power Plants***,* ***Gustavo García Martín****, M. de Miguel Gamo, M. Lasanta Carrasco, M. Lambrecht, F. Pérez Trujillo,* Universidad Complutense de Madrid, Spain

Zirconia-based sol-gel protective coatings have emerged as one of the most viable solutions to protect steels against molten salt corrosion in Concentrated Solar Power plants with nitrates as thermal energy storage. Thermal energy is used in the production of steam and electricity production subsequently, by means of the movement of a conventional turbine (Rankine cycle)

Central Tower design is a suitable technology to use new molten salts with higher thermal stability than nitrates, such as carbonates, which would allow plants to generate a higher amount of water vapour per unit of time, thus a higher conversion to electricity. Central receivers made of nickel-based material will increase the temperature from 565ºC with nitrates up to 800ºC with carbonates. Particularly, the outstanding eutectic ternary Li_2CO_3-Na_2CO_3-K_2CO_3 is presented as a real alternative nowadays. The drawback is its corrosion potential, hence protective coatings are being studied to enhance the endurance toward a levelized cost electricity (LCoE) reduction.

In support of these objectives, this work aimed at developing zirconia-based sol-gel protective coatings on the nickel-based alloy INCONEL 617 (Ni 53.28 wt.%, Cr 21.5 wt.%, Mo 8.80 wt.%) and the low chromo content alloy P91 (Cr 9.3· wt.%). Inconel would be used in high-temperature equipment such as receivers, exchangers, and high-thermal storage tanks. On the other hand, the ferritic-martensitic alloy might be used inlower-temperature parts, for instance, cold-temperature storage tanks, which could operate at 480ºC. Corrosion tests were performed at 480°C and 700°C up to 1000 h results were supported by gravimetric and microstructural characterizations. All results were compared to the uncoated steels. The results showed the promising behaviour of the coated substrates.

Coatings for Use at High Temperatures
Room Pacific E - Session A2-1-TuA

Thermal and Environmental Barrier Coatings I

Moderators: Dr. Sabine Faulhaber, University of California, San Diego, USA, **Dr. Kang N. Lee**, NASA Glenn Research Center, USA

1:40pm **A2-1-TuA-1 Influence of Microstructure on Phase Transformation of Plasma Sprayed YSZ Coatings Under Thermal Gradient Cycling Conditions***, **Simon Schöler**, D. Mack, Y. Sohn,* Forschungszentrum Juelich GmbH, Germany*; M. Rudolphi,* DECHEMA, Germany*; M. Adam,* TU Darmstadt, Germany*; R. Vassen, O. Guillon,* Forschungszentrum Juelich GmbH, Germany

The efficiency of gas turbines depends to a large extent on the gas inlet temperature. Thus, increasing the temperature capability of thermal barrier coatings (TBCs) represents a major scientific challenge, especially since segregation of yttria occurs in the t' phase of yttria partially-stabilized zirconia (8YSZ) at temperatures above 1250 °C in conventional use. For a more precise understanding of resulting critical phase transformations from tetragonal into the monocline phase, gradient tests were carried out on burner rigs in the present paper. The influence of different microstructures of YSZ TBCs on phase transformations at constant cooling rates in the range of 1-10 K/s from aging temperatures as high as 1600 °C were examined for two layer systems.

A porous YSZ layer was sprayed with high deposition efficiency using atmospheric plasma spraying (APS). A second layer system consisted of a porous APS-sprayed YSZ intermediate layer and a columnar YSZ topcoat, which was applied using suspension plasma spraying (SPS). Both systems were sprayed onto In738 substrates with bond coat. Due to the low corrosion resistance of the substrate at temperatures above 1300 °C, the TBC systems were tested in free-standing conditions in order to study in phase composition prior and after thermo-cyclic exposure at the high temperatures in comparison to former experiments conducted on substrate. For this purpose, the sprayed layers were detached from the bond coat using the method of electrochemical detachment and laser cut to the required sample geometry. The microstructures and phase composition of the free-standing specimen were characterized in the as-sprayed conditions and after the cycling tests using high-resolution scanning electron microscopy (SEM) and X-ray diffraction (XRD).

2:00pm **A2-1-TuA-2 A New Method to Diagnose Early Stages of CMAS Infiltration in Thermal Barrier Coatings***, **Vladimir Pankov**, K. Chen, P. Patnaik,* National Research Council of Canada

Siliceous particles such as dust, sand, volcanic ash, and runway debris represent a serious threat to hot section components coated with thermal barrier coatings (TBCs) in modern gas turbine engines operating at elevated temperatures. Due to a high content of calcium magnesium alumino-silicates (CMAS), these particles melt, adhere to the coating and infiltrate its open voids causing various modes of degradation. TBCs fabricated by electron beam physical vapor deposition (EB-PVD) are especially vulnerable to CMAS infiltration because of their columnar structure and open porosity. In this work a new non-destructive method has been proposed for detecting early stages of CMAS infiltration in TBCs and loss of their thermal stress tolerance. The method is based on analyzing the evolution of in-plane stresses in CMAS-infiltrated TBCs as a function of temperature using the $\sin^2\psi$ technique. The method was implemented in a modified industrial X-ray stress analysis facility. The TBCs with the composition of ZrO_2+7wt%Y_2O_3 were deposited on Hastelloy X substrates by EB-PVD and then infiltrated by volcanic ash from Mount Mazama. The TBC infiltration process was analyzed by weighting, scanning electron microscopy, and energy dispersive spectroscopy. The acquired experimental data demonstrated that visually undetectable amounts of CMAS result is a severe loss of thermal stress tolerance in EB-PVD TBCs while can be easily detected by the developed method. A model was proposed to explain the observed evolution of in-plane thermal stresses in TBCs infiltrated with different amounts of volcanic ash.

2:20pm **A2-1-TuA-3 Mechanical Behavior of a Nial Coating: Effect of Thermal Aging on the Brittle-to-Ductile Transition Temperature***, **Capucine Billard**, V. Maurel,* Mines ParisTech, PSL Research University, France*; D. Texier,* Institut Clement Ader (ICA), France*; D. Marquie,* Safran Aircraft Engines, France*; N. Bourhila,* Safran aircraft engines, France*; L. Marcin,* Safran aicraft engines, France

Nickel-based superalloys are high performance materials used for turbines blades. Due to the long duration of use of these structures and the complex thermomechanical load during a cycle of operations, a coating with an adequate resistance to corrosion and oxidation is essential. The material studied is a polycrystal nickel-based superalloy coated with an aluminure (NiAl) designs as a local aluminum reservoir to form a protective alumina film. NiAl coatings exhibit a brittle behavior up to 650-750°C. This brittle-to-ductile transition makes it sensitive to cracking during service, especially at low temperatures. Such cracking in the coating can lead to premature failure of the coated blade, then propagating under cyclic thermomechanical loading.

The objective is to link the microstructure specificities, with respect to the material's thermal history, to the mechanical response of the coating in temperature. Thermal treatment up to 1100°C, leads to progressively transform β-NiAl phase in γ'-Ni_3Al with the decrease of the Al content in the coating coupled with the diffusion of Ni from the substrate. It has been reported in NiPtAl coatings that thermal aging delayed the apparition of the first crack. This improvement in the ductility, observed at room temperature, is attributed to the γ' phase formation in aged specimens. From these results, our aim is to better understand how the brittle-to-ductile transition occurred in NiAl coatings and how thermal aging will impact it. The experimental approach is twofold. First, to study the crack onset, tensile tests have been carried out on dedicated specimens up to 900°C. The same protocol has been applied on thermal-aged specimens. Several thermal treatments conditions have been explored. As for NiPtAl coatings, results show a gain in ductility at room temperature. To assess the aging impact in the vicinity of the brittle-to-ductile transition temperature, attention has been paid on short time treatments. Mechanical results coupled with a detailed microstructure characterization from post-mortem materials will be presented. Then, micromechanical tests have been carried out using ultrathin bond-coating specimens, after thermal treatment or not. Indeed, due to the thickness of the coating, the direct thermomechanical measurement properties of the coating is not trivial. Assessing local properties within the gradient of microstructure may be useful for the understanding the effect of thermal treatment.

The presentation will compare the experimental results from these two campaigns to discuss the effect of thermal aging on the thermomechanical behavior of NiAl coating.

2:40pm **A2-1-TuA-4 Failure Mechanisms of Conventional Thermal Barrier Coatings and Development of Alternate Coating Systems for IGT Applications***, **Prabhakar Mohan**, B. Cottom,* Solar Turbines Inc., USA **INVITED**

Actively-cooled hot section components of industrial gas turbine (IGT) engines such as combustor liners, turbine blades and turbine stationary vanes continue to benefit from use of thermal barrier coatings (TBC). Zirconia stabilized with 7-8 wt.% yttria (YSZ) topcoat applied with an alumina-forming metallic bond coat system has been the TBC system of choice for many decades. An overview of successful long-term field experience of YSZ TBC from IGT components will be presented. In addition, life-limiting coating failure mechanisms documented for YSZ TBC applied by air plasma spray (APS) and electron beam physical vapor deposition (EBPVD) will be presented using field experience of stationary and rotating engine components such as combustor liners and turbine blades, respectively. Emphasis will be given to environmental degradation mechanisms of TBC by corrosive combustion byproducts as well as air-ingested CMAS-like deposits (calcium magnesium aluminosilicate sand). An overview of alternate TBC systems explored for lower thermal conductivity, higher temperature capability and / or enhanced thermal cyclic durability than conventional TBC will also be presented. Yttrium Aluminum Garnet (YAG) TBC applied by solution precursor plasma spray (SPPS) process demonstrated higher temperature capability and lower conductivity than APS YSZ TBC. In the case of EBPVD TBC, minor addition of a reactive element such as hafnium to platinum-modified diffusion aluminide bond coat demonstrated significant improvement in thermal cyclic durability of TBC when compared with baseline TBC bond coat. Ongoing coating testing and development efforts are targeted towards identifying promising lower conductivity (low-k) TBC systems for higher firing temperature applications.

4:00pm **A2-1-TuA-8 Manufacturing and Performance of a Three-Layer Environmental Barrier Coating System for SiC/SiC CMCs by Magnetron Sputtering***, **Ronja Anton**, V. Leisner, U. Schulz,* German Aerospace Center (DLR), Germany

When implemented in an aero-engine, SiC/SiC CMCs require environmental barrier coating (EBC) systems in order to withstand the atmospheric conditions. Physical vapour deposition processes enabled advanced design concepts of the EBC systems since its advantages lie within the precise control over microstructure and chemistry. A favourable EBC system consists of an Si-based bond coat followed by a rare earth, Y or Yb, disilicate as intermediate layer and finalised by a rare earth, Y or Yb, monosilicate top layer which protects the component against water vapour attack.

The EBC systems presented in this talk were manufactured by using magnetron sputtering for the bond coat and reactive magnetron sputtering for the intermediate and top layer. The X-ray amorphous coating system underwent a subsequent crystallization treatment. The macroscopic homogenous surface as well as local defaults like buckling or spallation of the silicate layers was defined during the crystallization process. Afterwards, the three layered EBC system was tested under cyclic oxidation at 1200 °C up to 1000 cycles where no major macroscopic changes appeared to the EBC system. For comparison, an uncoated SiC substrate and a SiC substrate with the bond coat as single layer were tested as well. The microscopic development in terms of phase stability, layer density and interface reactions were examined by SEM, XRD and TEM at different cycle numbers. While the bond coat and the disilicate remained hermetic dense, the monosilicate forms horizontal pores which agglomerated during long term testing. The disilicate and monosilicate remained phase stable while the bond coat started to form a SiO_2-based TGO layer on top. The oxidation kinetics of the bond coat within the EBC system was compared to the single layer bond coat during the thermocyclic testing. The behaviour of the EBC system in water vapour was investigated first as short-term experiment under aggressive conditions in 100 % water vapour steam with a gas velocity of about 10^{-1} m/s and secondly for a longer duration in about 30 % water vapour steam with a gas velocity of about 10^{-2} m/s at 1200 °C. The degradation behaviour was analysed and correlated to chemistry and morphology of the PVD coatings.

4:20pm **A2-1-TuA-9 EBC Multi-Layer Coatings on SiC-CMC Substrates Synthesized in a Continuous Vacuum Deposition Process*, Xavier Maeder,*** *D. Casari,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland*; D. Chen,* Oerlikon Metco (US) Inc., USA*; K. Glaentz,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein*; J. Michler,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland*; H. Schoech, B. Widrig, J. Ramm,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein

The mechanical stability at high temperatures of Ceramic Matrix Composites (CMC) material based on SiC compounds in combination with low density is the key concept to replace the Ni-based superalloys utilized in today's aircraft engines. However, the surfaces of these materials react with high pressure water vapour at high temperatures and volcanic ash (CMAS) and show instability due to volatilization, oxidation and diffusion. We present the capabilities of using a combined PVD-CVD technology in a continuous vacuum process to produce Environmental Barrier Coatings (EBC) to stabilize the surface of the CMC against chemical erosion processes and structural degradation. The approach for the coating design is based on a combination of adhesion layer and a chemical barrier dedicated for the CMC for the conditions of application in the turbine. Results for Si bond coat and Yb2Si2O7 barrier coating will be presented, with tests in water vapor up to 510h at 1316°C. SEM, TEM and XRD investigations were done to fully characterize the TGO growth and the diffusion along the interfaces. In addition to Si bond coat, other thin film interfaces have been tested in annealing experiments up to 1400°C in vacuum and in water vapour at 1316°C.

4:40pm **A2-1-TuA-10 Developments of the Slag-Based Geopolymer Coatings by the Flame Spray*, Wan-Ting Huang,*** *I. Huang, W. Lee, Y. Yang,* Department of Material and Mineral Resources Engineering, National Taipei University of Technology, Taipei, TAIWAN

Geopolymer materials are an emerging environmentally friendly material, due to the easy availability of raw materials, low carbon emissions and good physical properties such as high strength, fire resistance and thermal insulation properties, corrosion resistance, durability, etc. Powder application alkalization to increase its fluidity and reactivity, resulting in a powder form suitable for flame spraying. Therefore, in this experiment, the surface modification technology of flame spraying was used to prepare geopolymer coating on the surface of 304 stainless steel substrate to improve the corrosion resistance and fire resistance of the substrate, and the geopolymer was tested by salt spray test and fire resistance test. The resistance of the coating in high temperature or corrosive environments and the ability to protect the substrate. The experimental results show that the geopolymer coating has no cracks and large-scale spalling after the salt spray test for 7 days, which proves that the geopolymer coating has corrosion resistance and protects the substrate as the coating thickness increases. the better the effect; After 30 minutes of fire damage, the geopolymer coating did not peel off or burn, and the heating curve and the highest temperature measured on the substrate surface (temperature measuring surface) were significantly lower than those without coating. The exposed substrate of the coating proves that the geopolymer coating has the effect of fire resistance and thermal insulation. Therefore, the application of geopolymers material to the protective coating of steel components has great potential for development.

5:00pm **A2-1-TuA-11 Thermal Spray Coating with Ceramic Microspheres for Acoustic Absorption Applications*, Ting-Ya Chuang,*** *W. Lee, Y. Yang,* National Taipei University of Technology, Taiwan

Several studies point out that noise contributes to hearing impairment and poor mental health. With the advancement of technology, the continuously increasing noise is unbearable, and it needs to be solved. To solve the noise problem, this research uses the technique of thermal spray to prepare the polyethylene coatings with different concentrations of ceramic microspheres, which we expect to increase the noise immunity.

The results show that the noise immunity of the coatings and substrate varies at different frequencies. Ceramic microspheres in the polyethylene coatings makes structural of coating changes, and the structure, the thickness and the mass density will affect the noise immunity. This research shows that the polyethylene coating with ceramic microspheres can make the higher sound transmission loss effect than the polyethylene coating without ceramic microspheres, because ceramic microspheres change the structure of coatings.

Coatings for Use at High Temperatures
Room Pacific E - Session A2-2-WeM

Thermal and Environmental Barrier Coatings II

Moderators: Dr. Vladislav Kolarik, Fraunhofer Institute for Chemical Technology ICT, Germany, **Dr. Pantcho Stoyanov**, Concordia University, Canada

8:00am **A2-2-WeM-1 On the Suitability of MoNbTaW Based Thin Films to Act as Diffusion Barriers**, ***Georg C. Gruber***[1], Montanuniversität Leoben, Austria; *A. Lassnig, S. Zak,* Austrian Academy of Sciences, Austria; *M. Kirchmair,* Montanuniversität Leoben, Austria; *S. Wurster, C. Gammer, M. Cordill,* Austrian Academy of Sciences, Austria; *R. Franz,* Montanuniversität Leoben, Austria

To further improve the performance of diffusion barriers between Cu and Si, several new approaches were developed, such as the use of high entropy alloys (HEAs). HEAs are alloys with a configurational entropy of at least 1.5R, where R is the gas constant. On the one hand, HEA thin films can be deposited with amorphous microstructure which can be beneficial for their use as diffusion barriers. On the other hand, the lattice distortion within HEAs leads to an increase of the strain energy as well as the cohesion energy leading to an increased activation energy for the Cu diffusion. Within the current study, six different HEAs, based on the MoNbTaW system and alloyed with Ti, V, Cr, Mn, Zr or Hf, have been deposited by high power impulse magnetron sputtering (HiPIMS) as diffusion barrier layers between Cu and Si. The thickness of the HEA layers on the Si substrate was 20 nm, followed by a 150 nm thick Cu layer also deposited by HiPIMS. X-ray diffraction (XRD) investigations found that the MoNbTaW layers alloyed with Ti and V showed a body-centered cubic microstructure while the other alloys showed an amorphous microstructure. Subsequently, the bi-layers were annealed at temperatures ranging from 550 to 800 °C in a vacuum furnace. After annealing, the bi-layers were investigated by XRD, resistance measurement and laser-scanning-microscopy, to check for potential barrier failure. The lowest onset temperature for barrier failure of 600 °C was found for amorphous MoNbTaWZr, whereas the highest of 700 °C was observed for CrMoNbTaW. The obtained results are discussed in terms of strain and cohesive energy as well as the enthalpy of mixing between the fifth alloying element (Ti, V, Cr, Mn, Zr or Hf) and Cu. Within the study, the great potential of these HEAs for future diffusion barriers was demonstrated.

8:20am **A2-2-WeM-2 Improvement of EBC Performance by Controlling Driving Forces for Mass Transfers in Oxides**, ***Satoshi Kitaoka***, JFCC, Japan; *T. Matsudaira, T. Ogawa, M. Wada,* Japan Fine Ceramics Center, Japan **INVITED**

Environmental barrier coating (EBC) systems typically have a multilayer structure that consists of complex oxides such as silicates and aluminates to achieve the required performance through the use of layers with different characteristics. Such coatings with highly dense layers exhibit excellent gas shielding performance when they are exposed to a high oxygen potential gradient (dμO) at elevated temperatures.

In the case of Yb2Si2O7 (YDS) and Yb2SiO5 (YMS) layers that constitute the oxide-EBC systems, the application of dμO results in the inward diffusion of oxide ions and outward diffusion of Yb ions along grain boundaries (GBs), according to the Gibbs-Duhem equation. In the case of Al6Si2O13 (mullite) layer, inward GB diffusion of oxide ions and outward GB diffusion of Al ions occur. Cation transport induces decomposition of the complex oxides that comprise the layers, which leads to collapse of the multilayer structure. Hence, suppression of the outward diffusion of cations under dμO is extremely important in the design of robust EBC systems.

In this study, an EBC design to improve both the structural stability and environmental shielding properties of multilayer structures was investigated based on mass transfer mechanisms in the individual layers. The oxygen and cation fluxes at the outflow side in single-layer EBCs such as YMS, YDS, and mullite were significantly larger than those at the inflow side, in accordance with dominant cation transport under a high oxygen potential (μO) region near the surface, and dominant oxygen transport under the low-μO region in the vicinity of the interface between the oxide-EBC and Si-based bond coat or SiC/SiC substrate. This suggests that several different ions interdiffuse within multilayer EBC systems and can be separated into a single species according to the layer configuration by control of the GB densities and thicknesses of the layers. In addition, the interface between the YDS and YMS layers acts as an energy barrier for the outward GB diffusion of Yb ions. Therefore, the structural stability and oxygen shielding properties even for an common three-layer EBC system consisting of a YMS top layer, a YDS intermediate layer, and a mullite bottom layer are considered to be significantly improved by unifying the main diffusion species in each layer and simultaneously utilizing an energy barrier against the outward diffusion of cations.

Key Words: Diffusion, Grain boundary, EBC, Oxygen permeability, High temperature

9:00am **A2-2-WeM-4 Steam Oxidation Kinetics of Si / Modified $Yb_2Si_2O_7$ Environmental Barrier Coatings on SiC/SiC Ceramic Matrix Composites at 1250 °C – 1350 °C**, ***Kang Lee***, *J. Stuckner, M. Presby, B. Pulio,* NASA Glenn Research Center, USA; *W. Jennings,* HX5, USA

Environmental barrier coatings (EBCs) have enabled the implementation of SiC/SiC ceramic matrix composites (CMCs) in gas turbines by protecting CMCs from H_2O-induced volatilization. Improving the reliability of CMC components requires long-life EBCs and accurate EBC lifing. Steam oxidation-induced failure is the most frequently observed EBC failure mode. NASA previously reported that modifying Si / $Yb_2Si_2O_7$ EBC by adding Al_2O_3 or Al_2O_3-containing compound, such as mullite ($3Al_2O_3{\cdot}2SiO_2$) and YAG ($Y_3Al_5O_{12}$), in $Yb_2Si_2O_7$ reduces the EBC parabolic oxidation rates by up to about 20 times at 1316°C in steam. Oxidation is a thermally activated process and therefore the oxidation rates vs. temperature relationship typically follows the Arrhenius equation. This paper reports the results of follow-on studies that expanded the test temperature to 1250 °C and 1350 °C to understand the temperature dependency of oxidation rates, which is a key component for EBC lifing.

9:20am **A2-2-WeM-5 Oxygen Permeability, Failure Analysis and Life Prediction of Environmental Barrier Coatings Under Adverse Environments**, ***Prakash Patnaik***, Aerospace Research Centre, National Research Council Canada; *A. Kumar,* TECSIS Corporation, Canada; *K. Chen,* Aerospace Research Centre, National Research Council Canada

Environmental barrier coatings (EBCs) are typically used to protect ceramic matrix composites (CMCs) substrate against a harsh environmental attack such as high-temperature water vapour-induced recession in aero-engines achieving lower density, higher temperature tolerance capability and higher engine thrust-to-weight ratio. Under adverse service operations, the oxygen permeability of ytterbium disilicate (YbDS) topcoat and thermally grown oxide (TGO) silicon dioxide in EBCs plays a key role in determining EBCs durability and life span. Using physics-based model and thermodynamics calculations along with defect reaction formulae, oxygen permeabilities under dry oxygen and water vapour conditions, as well as different temperatures, partial pressures and top coat modifiers, are investigated. The results show that oxygen permeability for topcoat YbDS is an order of magnitude higher than for TGO, indicating that TGO hinders the oxidant diffusion stronger, proving to be the diffusion rate controlling layer. Moreover, water vapour strongly increases the oxidant permeation, with the defect reaction playing an important role. It is suggested that the mass transfer through the topcoat is primarily by outward ytterbium ion diffusion and inward oxygen ion movement, with the latter being dominant, particularly in water vapour environments.

The heterogeneous multi-layers designed for EBCs offer specific functions. High thermal strain and stress could develop due to materials mismatch and thermal gradient, leading to an accelerated degradation limiting EBCs durability and performance. Multi-physics/mechanics approaches were used to establish thermal and residual stress models for multi-layer EBCs considering the theory of composite elastic beam bending due to thermal gradient under force–moment equilibrium and stress-strain-curvature relationship. Residual stress distributions along the layer thickness were obtained for selected EBCs. Results are then compared with the test data, and the model exhibits a consistent variation in stress values across different coating layers. Specifically, thermal and residual stresses were evaluated within topcoats YbDS and ytterbium mono silicates (YbMS). Higher stress in YbMS makes it unsuitable as the topcoat despite its thermochemical compatibility between components and very low steam volatility. Finally, the lifetime prediction was explored for selected EBCs under specific water vapor contents and thermal cycle conditions based on the developed stress models.

[1] **Graduate Student Award Finalist**

9:40am **A2-2-WeM-6 Raman Spectroscopic Investigation of SiO_2 TGO Phase Transformation and Si and SiC Substrate Stress**, ***Michael J. Lance***, Oak Ridge Natinal Laboratory, USA; *M. Ridley, T. Aguirre, B. Pint,* Oak Ridge National Laboratory, USA

SiC ceramic matrix composites (CMCs) are desired for use in combustion environments to achieve higher turbine operating temperatures, although CMCs require environmental barrier coatings (EBCs) for protection from the gas environment. EBC systems are known to primarily fail through coating delamination via growth of a thermally grown oxide (TGO) at the EBC – silicon bond coat interface. The TGO undergoes a phase transformation during thermal cycling, which results in stresses that may encourage EBC spallation. Uncoated Si and SiC substrates were exposed to air and steam to form cristobalite TGOs of a thickness that remained intact upon cooling to room temperature. These TGOs were then thermally cycled across the phase transformation temperature for cristobalite and characterized with Raman microscopy to map the $\alpha \leftrightarrow \beta$ transformation. The stress in the silicon and SiC was simultaneously measured with Raman microscopy. Finite element modeling was used to predict the CTE mismatch stress and transformation stress in the TGO which was compared to the measured Raman stress. This research was funded by the Advanced Turbine Program, Office of Fossil Energy and Carbon Management, U.S. Department of Energy.

11:00am **A2-2-WeM-10 Hot Section Coating Technology as an Enabler for Sustainable Propulsion**, ***Eli Ross***, Pratt & Whitney, USA **INVITED**

Continued growth within the aviation sector is often seen as running counter to the shared objective of carbon-neutral flight. That growth trend brings not only higher demand and corresponding emissions, it also brings exposure to more aggressive and challenging operating environments for gas turbine engines. Accordingly, sustainable propulsion systems must build on recent learning from harsh environmental exposure while provisioning for a future state of more efficient, lower carbon engine technology sets encompassing a suite of higher thermal efficiency, hybrid-electric, and alternate fuel solutions. A common thread across this complex future turbine engine landscape is the need for continued improvement and development of thermal and environmental barrier coatings used in the hottest areas of the engine to enable both performance and durability. An overview and historical perspective of Pratt & Whitney work in this area will be presented, along with a system-level vision for the continued role of coating development in helping to deliver on a more sustainable net-zero future.

11:40am **A2-2-WeM-12 Development of Tantalum Coating by the Cold Spray**, ***Sheng-Wei Zeng***, Department of Material and Mineral Resources Engineering, National Taipei University of Technology, Taipei, Taiwan; *Y. Chung, W. Li,* National Chung Shan Institute of Science and Technology, Materials and Electro-Optics Research Division, Long-tan, Taiwan; *Y. Yang,* Department of Material and Mineral Resources Engineering, National Taipei University of Technology, Taipei, Taiwan

In the coating technology, cold spray(CS) is different from the traditional thermal spraying technology. The cold spray coating is formed by plastic deformation without high temperature melting, which can keep the original characteristics of the material during the spraying process and have a denser coating. Refractory metals, tantalum(Ta), has good performance at high temperature, used in the electronics industry, aviation, defense and medical treatment. This study used cold spray to prepare refractory metals, tantalum(Ta) coating, discusses the cold spray under different process parameters (chamber pressure, chamber temperature, working distance) affected the microstructure changes of the coating and its mechanical properties, such as porosity, hardness, tensile strength, etc.

Coatings for Use at High Temperatures
Room Golden State Ballroom - Session AP-ThP

Coatings for Use at High Temperatures (Symposium A) Poster Session

AP-ThP-1 Thermal Stability of Thick α- and γ-Al_2O_3 Coatings Deposited by High Speed PVD, *K. Bobzin,* ***Christian Kalscheuer****, M. Moebius, P. Hassanzadegan Aghdam,* RWTH Aachen University, Germany

Metal oxide coatings such as α- and γ-Al_2O_3 are state of the art for different high-temperature applications T > 800 °C. However, synthesis of stable α-alumina coatings is limited to chemical vapor deposition (CVD), due to its high formation temperature T >1000 °C. Physical vapor deposition (PVD) can realize deposition of aluminium oxide coatings at lower process temperatures T ≤ 600 °C. Nevertheless, metastable or amorphous Al_2O_3 phases can be formed in this case. This leads to a lower thermal stability and limits the application of these coatings at higher temperature. However, previous studies showed that High Speed PVD can realize the synthesis of cristalline α- and γ-Al_2O_3 coatings with high coating thickness of s > 10 µm at T ≈ 780 °C. In this study the thermal stability of α- and γ-Al_2O_3 coatings deposited by means of HS-PVD was investigated. High temperature (HT)X-ray diffraction (XRD) measurements were performed in order to study the phase transformations of the coatings as a function of temperature between T = 700 °C to T = 1,200 °C in vacuum and in atmosphere. Moreover, morphology, indentation hardness H_{IT} and indentation modulus E_{IT} of the coatings were investigated after HT-XRD. HT nanoindentation (HT-NI) measurements were performed to in-situ analyze the indentation hardness H_{IT} and indentation modulus E_{IT} at T ≈ 650 °C and the highest device measurement temperature of $T_{max,NI}$≈ 750 °C. The HT-XRD results show that up to a temperature of T = 1,200 °C neither in vacuum nor in atmosphere any phase transformations of coatings are recognizable. This confirms phase stability of the coatings up to this temperature. Moreover, the morphology of the coatings did not show any changes after HT-XRD, indicating a high structural stability. In addition, the morphology did not show any cracks detectable by scanning electron microscopy after HT-XRD. In situ HT-NI measurements showed a reduction of indentation hardness and indentation modulus compared to measurements at room temperature. However, the indentation hardness and indentation modulus after HT-XRD do not vary from the as-deposited state. The results of the conducted research reveal a high thermal stabillity of crystalline α- and γ-Al_2O_3coatings deposited by HS-PVD up to T = 1.200 °C. This can extend the application of these coatings to higher tempertaures.

AP-ThP-3 e-Poster Presentation: High-Temperature Stability and Mechanical Properties of Non-Reactive PVD-Synthesized $MoSi_2$ Coatings, ***Sophie Richter****, A. Bahr, T. Wojcik,* Christian Doppler Laboratory for Surface Engineering of high-performance Components, TU Wien, Austria*; O. Hunold,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein*; S. Kolozsvári, P. Polcik,* Plansee Composite Materials GmbH, Germany*; J. Ramm,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein*; H. Riedl,* TU Wien, Institute of Materials Science and Technology, Austria

Molybdenum disilicide belongs to the group of refractory transition metal silicides, which are highly attractive as oxidation-resistant materials to be applied in high-temperature regimes (i.e., above 1000 °C). The unique strength of $MoSi_2$ is based on its high melting temperature and the formation of a highly protective silicon-based oxide scale. In relation to the metallic and covalent bonding nature, $MoSi_2$ obtains unique mechanical properties, suggesting this disilicide as a promising candidate for future protective coatings.

Within this study, direct current magnetron sputtering (DCMS) as well as high-power pulsed magnetron sputtering (HPPMS) techniques have been deployed to grow $MoSi_2$ thin films. These coatings were non-reactively deposited in an in-house developed (laboratory-scaled) sputter system using 3’’ compound targets. The influence of the deposition parameters (e.g., substrate bias potential and deposition temperature) on the phase formation, morphology, chemical composition, and mechanical properties (e.g., hardness and indentation modulus) has been investigated systematically by high-resolution characterization methods such as X-ray diffractometry (XRD), scanning and transmission electron microscopy (SEM and TEM), energy-dispersive X-ray spectroscopy (EDS), as well as nanoindentation. Additionally, micro-cantilever bending tests have been performed to determine the fracture toughness of the as deposited thin films. Furthermore, we employed differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) to investigate the oxidation kinetics. The formed oxide scales have been analysed for different temperature regimes up to 1500 °C (1 hour in synthetic air). The samples were subjected to long-term oxidation treatments in ambient air at 1200 °C for up to 100 hours. Based on these results, sputter deposited $MoSi_2$ thin films constitute a promising protective coating material applied for challenging environmental conditions.

Keywords: Disilicides; PVD; Protective coatings; Oxidation resistance; Phase stability;

AP-ThP-4 Thermal Stability of Atmospheric Pressure Plasma Jet Deposited YSZ Top Coats and Sputtered Al Bond Coats on Inconel 617, ***Yung-I Chen****, L. Wang, X. Qiu,* National Taiwan Ocean University, Taiwan

In this study, microstructure and oxidation behavior of YSZ-alumina thermal barrier coatings after high-temperature annealing were explored. Ni-based superalloy (Inconel 617) was used as the substrate material. Al bond-coat was deposited by direct current magnetron sputtering. YSZ top-coat were deposited through several iterations of atmospheric pressure plasma jet deposition and rapid thermal annealing at 600°C. Multilayered YSZ/Al coatings were fabricated with various stacking periods. The Al sublayer transformed into a dense Al_2O_3 layer after annealing. The multilayered YSZ/Al_2O_3 coatings were further annealed at 1100 °C in ambient air. The YSZ coatings maintained at a tetragonal phase after annealing at 1100 °C for hundreds of hours.

AP-ThP-5 $Ti_{1-x}Al_xN$ PVD Coatings in Hot-Corrosion Environments, *O. Hudak,* ***Rainer Hahn****, A. Scheiber,* Christian Doppler Laboratory for Surface Engineering of high-performance Components, TU Wien, Austria*; L. Shang, O. Hunold,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein*; S. Kolozsvari,* Plansee Composite Materials GmbH, Germany*; H. Riedl,* Christian Doppler Laboratory for Surface Engineering of high-performance Components, TU Wien, Austria

Hot corrosion is an accelerated oxidation process commonly observed in high-temperature settings (650-950 °C), such as gas turbines, coal gasification plants, and waste incinerators. It is a phenomenon where sulfur-rich atmospheres (exhaust gases) react with salt impurities to form high-melting sulfate salts that adhere to machining components. There, the salt deposits elicit an accelerated degradation of the operating parts by forming porous, non-protective oxide scales, drastically reducing the longevity of in-service parts. Ni-, Co-, and Fe-based superalloys, representing the backbone in the previously mentioned applications, are especially prone to hot-corrosion attacks.

This contribution presents $Ti_{1-x}Al_xN$ as a potential candidate as a protective PVD coating for hot-corrosion environments. $Ti_{1-x}Al_xN$ coatings with varying metal content ratios were arc-evaporated on a Ni-based superalloy and tested in an in-house built hot-corrosion testing rig. By applying a sulfate-salt mixture from the alkali and alkaline earth metal groups, we tested coated and uncoated samples in a SO_x-rich atmosphere at 700 and 850°C for a maximum duration of 30 h and subsequently analyzed using a set of high-resolution characterization techniques.

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