Coatings for Use at High Temperatures
Room Sunrise - Session A2-1

Thermal and Environmental Barrier Coatings

Moderators: Lars-Gunnar Johansson, Chalmers University of Technology, Sweden, Kang Lee, NASA Glenn Research Center, USA

2:10pm **A2-1-3 Property Comparisons of Air Plasma Sprayed and Dense Homogeneous Yttrium Disilicate***, **Cory Parker**, R Golden, E Opila,* University of Virginia, USA

Modeling efforts for Rare Earth (RE) silicate Environmental Barrier Coatings (EBCs) typically utilize properties of dense homogeneous materials, yet EBCs are often processed using an Air Plasma Spray (APS) process which results in heterogeneous phase distribution, porosity, and splat microstructures. In this paper, properties of dense homogeneous yttrium disilicate processed by Spark Plasma Sintering (SPS) are compared to those of APS yttrium disilicate. Specifically, phase stability during thermal cycling, thermal expansion, and stability in high temperature steam are compared. Phase changes during repeated temperature cycling were characterized with X-ray diffraction. APS phase mixtures evolved during cycling whereas SPS yttrium disilicates were more phase stable. Thermal expansion of both materials was characterized up to 1400°C using dilatometry. Coefficients of Thermal Expansion (CTE) for APS yttrium disilicate varied significantly without a prior annealing treatment. Once annealed, the CTEs for the SPS material were significantly more consistent than those of the APS material. The silica depletion depth for yttrium disilicate samples was measured after steam-jet exposures at 1200°C for times of 60 to 250 hours and steam velocities between 150-180 m/s. The silica depletion rate was lower in dense homogeneous SPS yttrium disilicate compared to the more porous and heterogeneous APS materials. Cross sectional analysis of APS specimens showed both silica depletion to greater depths at splat boundaries and localized yttria-rich areas with minimal silica depletion. The implications of the heterogeneity of APS RE silicate phases and microstructures for EBC applications will be discussed.

2:30pm **A2-1-4 Performance of Vacuum Plasma Spray Bond Coatings***, **Michael Lance**, A Haynes, B Pint,* Oak Ridge National Laboratory, USA

The effects of composition, temperature and environment on the performance of vacuum plasma sprayed (VPS) NiCoCrAlYHfSi bond coatings (BC) on directionally-solidified (DS) 247 substrates with air plasma sprayed Y_2O_3-stabilized ZrO_2 has been investigated. Four compositions were investigated including the base composition and additions of Ti, B and Ti+B. The addition of B did not improve the average coating lifetime in 1-h cycles at 1100 °C in air with 10% H_2O in either case, however, the addition of Ti caused a decrease in lifetime. Photo-stimulated luminescence spectroscopy was used to map residual stresses in the thermally-grown Al_2O_3 scale. To study performance near the operating temperature of BCs in land-based turbines, the effect of water vapor was studied at 900 °C in laboratory air and air with 10% H_2O for 10-500 h cycles. Water vapor had little effect on the measured parabolic rate constants at 900 °C and a comparison of the oxide microstructures will be reported.

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2:50pm **A2-1-5 Predicting Microstructural Evolution in Aluminide Coatings during Manufacturing and Degradation in Service***, **Rishi Pillai**, A Chyrkin, T Galiullin, W Leng, D Grüner, D Naumenko, W Quadakkers,* Forschungszentrum Jülich GmbH, Germany **INVITED**

Protective metallic nickel aluminide (NiAl) diffusion coatings enhance the oxidation and corrosion resistance of the underlying high temperature materials. Aluminising provides a cost effective alternative and allows coating of complex shaped components. However, the composition of the substrate and the specimen geometry influence the final coating microstructure during the aluminising process and govern the high temperature behaviour of the coating system during subsequent exposure.

The formation of a protective alumina scale and the diffusion from the coating into the substrate result in the loss of Al and thereby the dissolution of the ß-NiAl phase in the coating. The extent of interdiffusion depends on the coating and substrate composition. The compatibility of a given type of a coating with its base material substantially influences the performance of a coated material during service. Potentially detrimental precipitate phases may form especially in the interdiffusion zone (IDZ) during the aluminising process and during subsequent service depending on the composition of the base material. Evaluation of the material's high temperature behaviour requires extensive experimental testing. Computational methods can substantially reduce the extensive experimental efforts required for coating evaluation and qualification.

In the current work, an in-house developed coupled thermodynamic and kinetic computational model was employed to predict the microstructural evolution in nickel aluminide (NiAl) diffusion coatings on Ni-base wrought and cast alloys during the coating (aluminising) process as well as during the subsequent high temperature service. Three Ni-base alloys (602 CA, IN718 and Rene80) were chosen to evaluate the influence of specimen thickness and alloy composition on the performance of nickel aluminide (NiAl) coatings. Specimens of different thicknesses (0.1-2 mm) were coated with nickel aluminide via chemical vapour deposition (CVD) on 602 CA and IN718 and via a slurry coating process on Rene80. The specimens were discontinuously exposed for 1000 h at 1100 °C in laboratory air. Specimens were removed from the furnace at 100, 300 and 1000 h.

Element concentrations and phase distribution were obtained by scanning electron microscopy (SEM). Phases were identified by energy/wavelength dispersive X-ray spectroscopy (EDX/WDX) and electron backscatter diffraction (EBSD). The modelling results were validated with experimental data. The computational approach assists in estimating the lifetime of the coating and provides a tool to predict microstructural changes in coating systems as a function of alloy/coating composition, time and temperature.

3:30pm **A2-1-7 Engineered Architectures of Gadolinium Zirconate/YSZ based TBCs Subjected to Hot Corrosion Test***, **Satyapal Mahade**,* University West, Sweden*; K Jonnalagadda,* Linköping University, Sweden*; N Curry,* Treibacher Industrie AG, Austria*; N Markocsan, P Nylén,* University West, Sweden*; R Peng,* Linköping University, Sweden*; X Li,* Siemens Industrial Turbomachinery AB, Sweden

Gadolinium zirconate (GZ) is considered as a promising top coat candidate for high temperature TBC applications. Suspension plasma spray has shown the capability to generate a wide range of microstructures including the desirable columnar microstructure. In this study, a triple layered TBC comprising a thin YSZ base layer beneath a relatively porous GZ intermediate layer and a denser GZ top layer was deposited by axial SPS process. Additionally, a blend TBC double layered architecture of GZ and YSZ comprising a thin YSZ base layer and GZ+YSZ mixed ceramic top layer was deposited by SPS. SEM analysis of the cross section in both layered and blend architectures revealed a columnar microstructure. The porosity content of the deposited TBCs was measured using image analysis and water intrusion method. The as sprayed TBCs were exposed to corrosive salt environment which comprised of a mixture of vanadium pentoxide and sodium sulphate at 900°C for 8 hours. The top surface and cross section of the TBCs after the hot corrosion test were analyzed by SEM/EDS. The phase content in the as sprayed and corroded TBCs were also analyzed by XRD.

3:50pm **A2-1-8 Thermal Barrier Coatings: The Next Generation***, **Maurice Gell**, E Jordan, R Kumar,* University of Connecticut, USA*; C Jiang, J Wang, B Nair,* HiFunda LLC, USA

Thermal barrier coatings (TBCs) must exhibit a multitude of properties to be successful. Critical properties include: high temperature phase stability, low thermal conductivity, sinter resistance (retention of low thermal conductivity) and thermal cycle durability. It has recently been shown that yttrium aluminum garnet (YAG) TBCs made by the Solution Precursor Plasma Spray (SPPS) process can potentially be used at temperature 200oC higher than state-of-the-art YSZ TBCs. To further improve the properties of SPPS YAG TBCs, emphasis has been given to greater reductions in thermal conductivity by introducing linear arrays of porosity, called inter-pass boundaries (IPBs), and by demonstrating that thicker coatings with adequate durability can be fabricated. This presentation will present the results of these efforts and the associated thermal cycling durability.

4:10pm **A2-1-9 Microstructure of Gas Flow Sputtered Thermal Barrier Coatings***, **Nils Rösemann**,* Institute for Materials, TU Braunschweig, Germany*; K Ortner,* Fraunhofer Institute for Surface Engineering and Thin Films IST, Germany*; M Bäker,* Institute for Materials, TU Braunschweig, Germany*; J Petersen,* Fraunhofer Institute for Surface Engineering and Thin Films IST, Germany*; G Bräuer,* Institute of Surface Technology, TU Braunschweig, Germany*; J Rösler,* Institute for Materials, TU Braunschweig, Germany

Thermal barrier coatings (TBC) reduce the thermal load of gas turbine components. State-of-the-art TBCs consist of partially yttria stabilized zirconia (PSZ) and are deposited by thermal spray techniques (e. g. atmospheric plasma spraying) or electron beam physical vapor deposition (EB-PVD). The resulting microstructure is dependent on the process and strongly influences the coating´s properties and time to failure. Columnar structures exhibit a higher lifetime and are thus favored.

This talk investigates an alternative deposition technique (gas flow sputtering - GFS), which also gives rise to columnar microstructures. The aim of this work is to gain a fundamental understanding of the influence of crucial process parameters such as substrate temperature and bias voltage on the microstructure, and the suitability of GFS coatings for the use as TBCs.

PSZ coatings were deposited on polished FeCrAlY-Alloy substrates and described utilizing WDX, SEM, FIB and XRD.

The substrate temperature has been identified to be a crucial parameter. Between 500 and 800 °C, four different types of columnar microstructures are defined based on XRD pattern and morphology. The growth direction of the columns changes from <111> to <100> accompanied by a change in column shape from triangular to four-sided. The principal shape of the columns is furthermore explained using a growth model.

Applying bias voltage at a given substrate temperature does not give rise to a new type of microstructure but alters the microstructure defined by the substrate temperature. While low bias values lead to more regular columns, high bias voltages lead to further densification and compressive stresses, rendering these conditions unsuitable for TBC manufacturing.

Concluding, promising microstructures are presented accompanied by general guidelines for the suitable process parameters.

4:30pm **A2-1-10 Current Environmental Barrier Coatings Research at NASA***, **Kang Lee**, D Waters,* NASA Glenn Research Center, USA

SiC/SiC Ceramic Matrix Composites (CMCs) are a game changer for advanced power generation equipment because of their high temperature capability, oxidation resistance, and light weight that translate to significant reduction in fuel consumption and pollution. Limitations of SiC/SiC CMCs include surface recession and component cracking and associated chemical and physical degradation in the CMC. The solution pursued to mitigate surface recession of SiC/SiC CMCs is the incorporation of coating systems that provide surface protection, which has become known as an Environmental Barrier Coating (EBC). Other key life-limiting EBC degradation modes include oxidation in steam environments, CMAS-EBC reactions, and thermal fatigue. The first and second generation EBCs developed in mid 1990s-early 2000s laid the foundation for current EBCs. Many engine tests have been conducted since late 1990s and a first CMC component entered into service in 2016 in a commercial engine. The introduction of CMCs represents significant challenges as failure of the EBC means rapid reduction in component life. A reliable life model and testing methods relevant to engine conditions to validate life model need to be developed. This paper will discuss the current research activities on EBC development and testing at NASA.

4:50pm **A2-1-11 CMAS Infiltration Prediction for 7YSZ TBCs Deposited by EB-PVD***, **Juan Gomez**,* The University of Texas at El Paso, USA*; R Naraparaju, U Schulz,* German Aerospace Center (DLR), Germany*; R Chintalapalle,* University of Texas at El Paso, USA

The current demand for higher gas turbine engine operating temperatures has brought into attention the hot corrosion attack to thermal barrier coatings (TBCs) due to siliceous debris infiltration into gas turbines. The debris are commonly composed of calcium-magnesium-alumino-silicates (CMAS) which are carried in environmental pollution in the form of sand, fly ash, volcanic ash, among others. Previous studies in standard 7YSZ coatings deposited by EB-PVD techniques have shown a significant relation of coating micro-structure and CMAS infiltration depth. In the previous study a physical model was proposed to estimate CMAS infiltration into EB-PVD coatings by simplifying the micro-structure as a concentric pipe (kernel pipe) with open channels (feather arms) which distribute the CMAS reducing the infiltration time into the TBC. Therefore, it is of highly importance to understand the kinetics of CMAS infiltration since by refining the deposition parameters (deposition pressure, rotation, etc.) CMAS infiltration can be reduced. The current work correlates experimental results obtained for short term CMAS infiltration performed at 5 min. using a synthesized CMAS source with theoretical results using the proposed "concentric pipe model". The calculated results are in good agreement by closely predicting the infiltration time for a given depth when compared with short term infiltration experiments. Additionally, the results were compared with the "open pipe" infiltration model which has been previously used in literature for CMAS infiltration estimation. The results also show a significant error in infiltration prediction for the open pipe model which proved a more realistic approach for CMAS infiltration using the proposed concentric pipe model.

5:10pm **A2-1-12 Oxidation Behavior of CrN, AlCrN, and AlTiN Cathodic Arc PVD Coatings***, **Zuhair Gasem**, A Adesina,* King Fahd University of Petroleum and Minerals, Saudi Arabia

The oxidation behaviors of CrN, AlCrN, and AlTiN *PVD-cathodic-arc* coatings were evaluated isothermally at 800°C, 900°C and 1000°C for a duration of 5hr in ambient atmosphere using 304 stainless steel substrates. A novel substrate design was employed where all substrate edges were chamfered at both faces to coat both surfaces and edges. The oxidation rate measurement of all-surface-coated samples were conducted using *in-situ* thermogravimetric apparatus. The coating composition, morphology, and hardness were analyzed before and after the oxidation test using X-ray diffraction (XRD), energy dispersive spectroscopy (EDS), and scanning electron microscope (SEM).

XRD results for CrN revealed the evolution of various temperature-dependent oxides forming within and beneath the coating including Cr_2O_3, Fe_2O_3, and Cr_2O after the oxidation tests. The diffraction peaks for CrN coating (1.8 um in thickness) disappeared entirely after the oxidation test at 1000°C suggesting complete transformation of the CrN layer into mixed oxide layer. The specific weight gain curves showed continuous increase with exposure time at all temperatures examined. The oxidation resistance of AlCrN coating exhibited significant improvement as compared to CrN coating as can be evident by comparing the oxidation kinetic curves of the two coatings owing to the formation of the more protective Al_2O_3 in addition to Cr_2O_3. The coating provided effective protection at 800°C and 900°C as can be inferred from the oxidation rate curves. The thickening of AlCrN layer, the absence of oxidation product at the interface between the coating and the underlying substrate, and the asymptotic oxidation rate behavior suggest persistence of protection of AlCrN coating up to 1000°C.

The major oxidation products of AlTiN coating over 900-1000°C comprised of TiO_2 and Al_2O_3. The oxidation kinetics showed initial rapid growth stage and a delayed linear behavior. The initial stage may be attributed to the formation of porous TiO_2 with non-continuous Al_2O_3. The delayed linear oxidation rate is similar to the oxidation rate of AlCrN coating at 1000°C. This has been explained in terms of the dominance of the growth of continuous Al_2O_3 layer in both coatings at high temperature.

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