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.

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