Coatings for Use at High Temperatures
Room Grand Hall - Session AP

Symposium A Poster Session

AP-1 Feasibility of using Rare-earth (La and Ce) Sulfates as Functional Embedding Agents for Thermal Barrier Coatings*, D Song, T Song,* Hanyang University, Republic of Korea*; **HyeonMyeong Park**, Y Jung,* Changwon National University, Republic of Korea*; J Zhang,* Indiana University Purdue University Indianapolis, USA

Feasibility of using rare-earth (La and Ce) sulfates as functional agent was demonstrated for thermal barrier coatings (TBCs). Both pellet and TBC specimens were prepared and intentionally cracked by indentation methods. Then an isothermal heat treatment for pellets and cyclic thermal fatigue (CTF) test for TBCs were performed. The phase transformation behaviors were investigated by X-ray diffraction and X-ray photoelectron spectroscopy. Rare-earth oxides and zirconates were formed during the heat treatment due to the high oxygen ion diffusivity of yttria-stabilized zirconia (YSZ) matrix. TBCs with rare-earth sulfates showed longer lifetime performance in the CTF test than typical YSZ TBC without functional agents. This is due to crack healing and inhibiting effects resulted from the reaction of the functional agents with YSZ matrix. This study and derived results demonstrate the applicability of rare-earth sulfates as functional embedding agent through further optimization of microstructure design and encapsulation process.

AP-2 Lifetime Performance of Yb-Gd-Y-based Thermal Barrier Coatings with Buffer Layer in Thermally Graded Mechanical Fatigue Environments*, **Bong-Gu Kim**,* School of Materials Science and Engineering, Changwon National University, Republic of Korea*; G Lyu, S Jung, H Park, Y Jung,* Changwon National University, Republic of Korea*; J Zhang,* Changwon National University, Republic of Korea, USA

The effects of buffer layer on the crack generation and thermal fatigue behaviors of Yb-Gd-Y-stabilized zirconia (YGYZ) based thermal barrier coatings (TBCs) were investigated through thermally graded mechanical fatigue (TGMF) test. Double buffer layers were introduced to enhance the thermomechanical properties in the YGYZ based TBC systems, deposited with the regular and high purity 8YSZ buffer layers. TGMF tests were performed at 1100 ^{o}C with a tensile load of 60 MPa, till 50% spallation of the top coat or cracking at the interface between the top and bond coats. The multilayer TBCs showed longer lifetime performance compared to the single layer YGYZ based TBCs, showing delamination and/or cracking at the interface between the buffer layer and the top coat in the multilayer TBCs. The feedstock purity in the buffer layer was also effective in enhancing the lifetime performance of YGYZ based TBC system in the thermal and mechanical environments. Failure mechanisms in the layered TBCs were investigated and discussed based on the crack initiation and propagation behaviors observed through the TGMF tests.

AP-3 Thermal Durability of Thermal Barrier Coatings – Effect of Purity and Monoclinic Phase in Feedstock Powder*, **Yeon-Gil Jung**, H Park, S Jeon, G Lyu, S Jung,* Changwon National University, Republic of Korea*; K Park, I Kim, B Yang,* Doosan Heavy Industries and Construction, Republic of Korea*; J Zhang,* Indiana University, USA

The effects of the purity and monocline phase of feedstock powder on the thermal durability in thermal barrier coatings (TBCs) were investigated through the jet engine thermal shock (JETS) test. Three kinds of feedstock powders, such as regular purity (YSZ), high purity (HP), and non-monoclinic phase (nMP) in 8 wt% yttria-stabilized zirconia, were deposited on the Ni-Co based bond coat by an air plasma spray (APS) process. The thicknesses of the top and bond coats were designed and controlled as 400 and 200 µm, respectively. In each cycle of JETS test, the top surface of TBC was heated with flame of 1400 °C for 25 s, and then cooled for 25 s using nitrogen gas. Regardless of feedstock species, all samples showed sound condition in the JETS test up to 2000 cycles. As the number of cycles increased over 2000, the lifetime of the TBC with HP powder was longer than those with the YSZ and nMP powders. The TBC with YSZ powder showed the shortest thermal durability in the TBC with YSZ powder. The relationship between feedstock species and thermal durability is extensively discussed, based on microstructure evolution and phase stability during the JETS tests.

AP-6 Integral vs. Local Chemical Composition of (coating) Materials: Is your Solid Solution a Solid Solution?*, **Jochen M. Schneider**,* RWTH Aachen University, Germany

Atom probe tomography provides the opportunity to investigate the chemical composition of (coating) materials on the nm-scale. During the multimedia poster session differences (and similarities) between the integral coating composition determined by elastic recoil detection analysis and/or energy dispersive X-ray spectroscopy and the local composition determined by atom probe tomography will be presented. The effect of Si additions to Cr_2AlC which are known to affect the oxidation kinetics are presented as well as Mn additions to Cr_2AlC. Furthermore, differences (and similarities) in local and global chemical composition identified for the coating systems Mo_2BC as well as transition metal nitrides and oxynitrides are discussed.

AP-7 Coating Generation and Study for Materials Protection used in Extreme Atmosphere: Sustainability and Energy Efficiency*, A Illana,* Universidad Complutense de Madrid, Spain*; M Gutiérrez, I Baraibar,* Instituto Nacional de Técnica Aeroespacial (INTA), Spain*; S Mato,* Universidad Complutense de Madrid, Spain*; R Muelas Gamo,* Instituto Nacional de Técnica Aeroespacial (INTA), Spain*; M Benito, A Bahillo,* Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas (CIEMAT), Spain*; **Francisco Javier Pérez-Trujillo**,* Universidad Complutense de Madrid, Spain*; A Agüero Bruna,* Instituto Nacional de Técnica Aeroespacial (INTA), Spain

The need to increase the efficiency in energy production in a sustainable way as well as reducing the environmental impact, has become evident. This leads to increasingly extreme conditions imposing more demanding performances on materials such as higher temperatures and pressures in more corrosive environments. Therefore, the main goal of "Coating Generation and Study for Materials Protection used in Extreme Atmosphere: Sustainability and Energy Efficiency" project is developing and optimizing protective coatings for structural materials of power plants generation to protect from aggressive environments present in power plants working under oxy-fuel atmospheres and employing biomass as fuel.

The technical goal aiming at studying the feasibility of using coatings as an alternative in a biomass oxy-combustion thermal plant working under supercritical conditions. Until now, it had been unexplored, so this project has assessed the corrosion behavior of bare and coated coupons of ferritic steels (T22 and P92) and a high-alloy multi-purpose austenitic stainless steel (Sanicro 28) which have been exposed to biomass oxy-combustion conditions on one side, and to supercritical steam on the other, both in laboratory and in pilot plants. The gravimetric evolution was analyzed at fixed intervals to study their oxidation kinetics. In addition, common characterization techniques have been used, such as x-ray diffraction (XRD), to explored the phases formed during the oxidation process, and scanning electron microscopy with energy dispersive detector (SEM-EDX), to evaluate the morphology and semi-quantitative composition.

On one hand, the samples were tested in pure steam at 650ºC and 0.1 and 30 MPa in order to evaluate the role of the pressure. After more than 1000 h, the coated materials exhibited an improved behavior compared with base materials. However, this parameter significantly affected the ferritic steels.

On the other hand, the oxy-combustion tests were firstly made in a laboratory with and without additions of K_2SO_4 and KCl and/or SO_2 in order to simulate the composition of thistle biomass, which has been used in experiments carried out after at the pilot plant. In all cases, ferritic steels have been severely affected, forming non protective oxides. Nevertheless, Sanicro 28 steel has been able to resist so it does not need to be covered. Regarding the coatings, they have successfully altered the catastrophic behavior of the ferritic steels. So it could contribute to reach the production of sustainable, clean and efficient electricity using existing infrastructures.

AP-8 The Influence of Reactive Elements on Thermogravimetric Behaviour of New Co-Ni-Al-W Superalloys Dedicated to Bond-coat Deposition*, G Moskal, A Tomaszewska, **Damian Migas**,* Silesian University of Technology, Poland

Characterization of as-cast microstructure and oxidation resistance of new type of cobalt based superalloys with γ/γ′ microstructure were presented in this investigations. As a referential material the Co-20Ni-7Al-7W (at.%) was used as the modified version of basic (Co-20Ni-7Al-7W). All investigated alloys were modified by addition of yttrium, hafnium and zirconium on the level of 0.5, 0.2 and 0.05 at.% respectively due to improvement of its oxidation resistance. All alloys were made in Institute of Materials Engineering of Silesian University of Technology in Poland.

The oxidation performance of all alloys were made during thermogravimetric investigations at temperature range from 25 to 1200°C. The main analyzed parameter was mass gain detected continuously during the test. After the test the overall and cross-sectional analysis of specimens was made and included analysis of oxide layer morphology on the surface of specimens with characterization of phase constituent of oxide layer. The detailed analysis of oxidized layer was made by scanning electron microscopy method when distribution of alloying elements was made with special attentions on yttrium localization after the test in oxide zone.

Obtained data showed that both addition of Y, Hf and Zr has a strong influence on oxidation performance of new Co based superalloys. Those influence is demonstrated mainly by different morphology of final oxide zone with strong segregations of yttrium containing oxidation products.

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AP-9 Study the Surface-aluminizing Coating to Enhance High-temperature Oxidation Resistance of T91 Boiler-used Steel*, **Wu Kai**, Y Chen, C Chung,* National Taiwan Ocean University, Taiwan

The oxidation behavior of surface aluminizing T91 boiler-used steel (T91A) was investigated over the temperature range of 600 ~ 900 ˚C in dry air. The surface-aluminizing parameters for T91A were to heat the T91 samples at 800 °C for 4 hr in 7% AlF_3 at a 200 cc/min flow rate of Ar. The results showed that the oxidation kinetics of T91A followed a single-stage parabolic-rate law at 600 ~ 750 ˚C, while a two-stage parabolic-rate law was observed at 800~900 ˚C. The steady-state oxidation rates at 850 ~ 900 °C were significantly slowly than those at lower temperatures. The oxidation rates of the aluminizing alloy were significantly lower than those of the T91 substrate by 6.2 orders of magnitude at 850 ˚C. Both α- and θ-Al_2O_3 formed on top of T91A surface after the oxidation. The amount of θ-Al_2O_3 gradually reduced and the amount of α-Al_2O_3 gradually increased with increasing temperature. It was found that the growth of α-Al_2O_3 is strongly dependant on the oxidation temperature and duration of time. The formation of α-Al_2O_3 is responsible for the lower oxidation rates of the T91A alloy at 850 ~ 900 ˚C.

AP-10 New Insights into the Oxidation Behaviour of AlCrSiN Coatings and an Approach to Avoid Trans-interface Diffusion at Elevated Temperatures*, **Nikolaus Jäger**, S Klima, M Meindlhumer,* Montanuniversität Leoben, Austria*; H Hruby,* eifeler-Vacotec GmbH, Germany*; J Keckes, R Daniel,* Montanuniversität Leoben, Austria

Applications such as high-speed machining and dry cutting of metals demand the development of superior materials to withstand severe operating conditions. High loads and temperatures exceeding 1000°C in contact between the tool and the work piece require protection of the tool surface by advanced hard coatings. Under such conditions, thermal stability and oxidation resistance of the coated tool is of outmost importance. A solid understanding of the diffusional mechanisms resulting in the phase transformation of metastable phases and oxidation of the coating with subsequent deterioration of the mechanical properties is thus the basis for establishing strategies for improved high-temperature behaviour of the protective coatings. The coating performance may also be dramatically affected by structural changes driven by elements diffusing from the tool material into the coating.

In this work, AlCrSiN coatings with a Si content up to 10 at. % were deposited on cemented carbide and high-speed steel substrates by cathodic arc evaporation. To study the oxidation behaviour, the samples were annealed in ambient atmosphere at temperatures up to 1200 °C for 1 h. Powders made out of the coatings were investigated via differential scanning calorimetry and thermogravimetry. Additionally, an $AlCrSi_{10}N$ coating on cemented carbide annealed at 1400 °C for 1 h was characterized by cross-sectional, position-resolved synchrotron X-ray nanodiffraction analysis to gain information about structure, texture and residual stresses across the coating thickness including the oxide scale formed on its surface. To investigate the influence of diffusion of substrate elements into the coating, samples were annealed in vacuum in the same temperature range and analysed by means of scanning electron microscopy and energy dispersive X-ray spectroscopy. Additionally, one selected sample was characterized by cross-sectional, position resolved synchrotron X-ray nano-diffraction analysis to study the microstructural evolution, phase transformations and development of residual stresses near the substrate-coating interface affected by pronounced diffusion of substrate elements.

Based on the results of this study, we will suggest strategies to suppress diffusion of Co from the cemented carbide and Fe from the steel substrates into the coating and enhance the oxidation resistance of AlCrN coatings to ensure high-temperature stability of the coating and thus enhanced operation performance of the coated tool.

AP-12 Gradient SiBCN Ceramic Coating for High-temperatue Anti-oxidation Protection of Carbon-carbon Composite*, **Zongbo Zhang**,* Institute of Chemistry, Chinese Academy of Science, China

Carbon-carbon(C/C) composites , an important high-temperature structural material, face the severe problem of oxidation only above 500℃. Coating technology with ultra-high temperature ceramics (UHTCs) have been proved to be highly effective to improve the oxidation resistance of C/C composites[1]. Currently, the main coating techniques for C/C composites include pack cementation, plasma spraying, polymer derived ceramic coatings and so on. Among them, polymer derived ceramic coatings have gained more and more attention due to its easy for implementation, no need for special equipment and low sintering temperature. However, the technique of polymer derived ceramic coating usually needs multiple casting-sintering cycles to densify the prepared coating, which requires ultra long operation time thus affect its real application. In this paper, we present a novel strategy to fabricate gradient SiBCN composite coating with just one casting-sintering cycle by adopting polyborosilazane ceramic precursor[2] as the main raw material, well controlling the viscosity of the polyborosilazane-filler slurry and a low temperature pre-oxidation treatment.

The microstructure morphology, elements composition, thermal performance, and anti-oxidation property of the ceramic coating have been investigated. The coating surface macro appears uniform, no cracks, while microscopic loose and porous. The coated C/C composite exhibits excellent anti-oxidation property with weight loss of only 0.06% after oxidation at 1500 C for 30min. Due to the gradient structure, the coating shows no crack and no peeling off after 8 cycles of thermal shock from 1500°C to room temperature. The XRD analysis showed that the surface of the coating was oxidized to form a zirconium silicate material, thereby preventing oxygen from further penetrating and achieving the oxidation resistance. All above of the results indicate that SiBCN ceramic coating has high potentials for anti-oxidation protection of C/C composites.

Reference

[1] Davies I J, Rawlings R D.(1994) ,Microstructural investigation of low-density carbon-carbon composites, *J. Mater. Sci.*, 29, 338-344.

[2] Zongbo Zhang, Caihong Xu.(2011), Synthesis and characterization of a new liquid polymer precursor for Si–B–C–N ceramics, *J Mater Sci.*, 46,5940–5947.

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