Coatings for Use at High Temperatures
Room Royal Palm 1-3 - Session A2

Thermal and Environmental Barrier Coatings

Moderators: Kang Lee, NASA Glenn Research Center, USA, Lars-Gunnar Johansson, Chalmers University of Technology, Sweden, Pantcho Stoyanov, Pratt & Whitney, USA

8:00am **A2-1 Corrosion Degradation of High Temperature Coatings: Similarities and Differences for Marine and Aero-Turbine Applications***, **Daniel Mumm**,* University of California, Irvine, USA **INVITED**

Gas turbine engines are utilized throughout the world, as aircraft and ship propulsion systems and as stationary power generation systems. The materials utilized in the hot-section of marine gas turbines are subject to a wide range of high temperature oxidation/corrosion degradation processes, dependent upon the fuels used and the site-specific environments. Operation in marine environments leads to salt-laden intake air, whereas additional constituents in the intake air (pollutants, atmospheric particulate matter, etc.) have unique influences on the oxidation/corrosion degradation mechanisms of thermal and environmental barrier coating utilized with hot-section components. Ongoing research has also illustrated that the performance and lifetime of such materials is also highly dependent upon the exposure temperatures and the specific thermal cycling history associated with operational service demands. This talk will discuss ongoing efforts to explore high temperature oxidation and corrosion processes, and the resulting materials degradation, under more complex exposure conditions – with a focus on the synergistic role of mixed-mode (Type II, Type I and oxidation condition) exposures and complex corrosive deposit chemistries including sulfate mixtures and oxides. The research findings are discussed in relation to the performance of marine propulsion, aero-propulsion and power-generation turbines operating in marine environments. Implications for understanding of the observed degradation mechanisms is essential for enabling the design of materials capable of being utilized in increasingly hostile gas turbine operational scenarios.

8:40am **A2-3 Evolution of Microstructures and Interfaces in Doped, Layered, and Composite Coatings Exposed to Sand Laden Flows in a Gas Turbine Engine***, **Andy Nieto**, M Walock, A Ghoshal, M Murugan,* US Army Research Laboratory, USA*; D Zhu,* NASA Glenn Research Center, USA*; W Gamble, J Swab, B Barnett, M Pepi,* US Army Research Laboratory, USA*; R Pegg, C Rowe,* US Navy Naval Air Systems Command, USA

Calcia-magnesia-alumina-silicate (CMAS) attack on thermal barrier coatings (TBCs) have led to a need for new TBC coating designs that incorporate mechanisms for mitigating CMAS deposition and infiltration, while maintaining or enhancing the critical properties of TBCs, namely durability and strain tolerance under high temperature and velocity in cycling engine operating conditions. Several approaches have been investigated here, including the use of dopant elements and phases, multi-layered coatings, and blended composite coatings. These included doped ZrO_2 based coatings, layered Y_2O_3 stabilized ZrO_2 (YSZ), HfO_2, and Gd_2O_3 coatings, as well as composite YSZ/Gd_2O_3 coatings. The coatings were deposited on turbine nozzle vanes and tested inside of a full scale engine with air flow temperatures at ~1400 °C and several sand ingestion cycles. SEM, EDS, and TEM were utilized to compare the microstructures before and after exposure to the sand laden combustion flows within the gas turbine engine. Emphasis was placed on the interfaces formed between the coatings and the formed CMAS deposits. Interfaces within the coatings, such as those in between phases or between layers, were also evaluated in order to understand which microstructural designs were most effective at mitigating CMAS infiltration/attack and particulate adhesion, whilst maintaining structural stability and integrity.

9:00am **A2-4 The Effect of HVOF Bond Coating with APS Flash Coating on TBC Performance***, **Michael Lance**, J Haynes, B Pint,* Oak Ridge National Laboratory, USA

Previous work reported the benefit of a ~50 µm thick air plasma sprayed (APS) "flash" coating to improve the performance of high-velocity oxygen fuel (HVOF) bond coatings. In this study, NiCoCrAlYHfSi HVOF bond coatings were deposited on directionally-solidified (DS) 247 disk substrates with APS yttria-stabilized zirconia (YSZ) top coatings. The HVOF-only bond coatings were compared to APS flash coatings of NiCoCrAlYHfSi and NiCoCrAlY using 1-h cycles at 1100 °C in air with 10% H_2O. Photo-stimulated luminescence spectroscopy was used to map residual stresses in the thermally-grown Al_2O_3 scale as a function of exposure time. Five specimens of each coating type were cycled to determine an average coating lifetime under these conditions and, after failure, oxide thickness and morphology were compared on various bond coatings to better understand the influence of the flash coating presence and composition.

Research sponsored by the U. S. Department of Energy, Office of Fossil Energy's Turbine Program.

9:20am **A2-5 Influence of Process Conditions and Ceramic Doping on the Performances of Advanced TBCs Based on Al Slurry***, **Germain Boissonnet**, B Grégoire, J Balamain, G Bonnet, F Pedraza,* University of La Rochelle, France

Current processes of fabrication of thermal barrier coatings (TBCs) for the hottest sections of aeronautical engines are very complex and quite expensive. Therefore, they cannot be applied to other sections that require thermal insulation due to the increase of the turbine inlet temperatures. Among the alternative coating techniques, slurries from Al microspheres that result in the formation of hollow alumina top foam appear particularly attractive [1-3]. Their insulation properties are indeed equivalent to those of conventional APS YSZ coatings [4]. However, the current process of fabrication leads to poor mechanical resistance of this light foam [5].

Therefore, the effects of the annealing atmosphere and of the introduction of Al_2O_3 and YSZ ceramic particles on the strengthening and on the thermal insulation potential of the ceramic foam will be presented. Alumina was chosen due to its good chemical compatibility with the Al slurry to harden the top coat while binding with the hollow oxide spheres. Concurrently, YSZ is expected to lower the overall thermal conductivity of the top coat while simultaneously increasing its stress compliance *because of the high thermal expansion coefficient of this material*. Low water vapor pressures will also be shown to thicken the shells of the hollow particles compared to Ar and oxygen [5]. Also, the reaction mechanisms of YSZ particles with molten Al to result in the formation of $Al_3Zr/\alpha\text{-}Al_2O_3$ composites [6] that leads to remarkable changes in thermal and mechanical properties of the top coat will be described in light of the results from DSC, XRD, Raman micro-spectrometry and thermal diffusivity.

References

1. F. Pedraza et al., Materials Chemistry and Physics 134 (2012) 700-705.

2. X. Montero et al., Surface & Coatings Technology 206 (2011) 1586-1594.

3. V. Kolarik et al., Materials at High Temperatures 29, *2* (2012) 89-94.

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6. H. Zhu et al., Composites Science and Technology 70 (2010) 2183-2189.

9:40am **A2-6 Synthesis and Characterization of Combined Oxides and Ni Superalloy Coatings by Cathodic Arc Evaporation for Bond Coat Application***, X Maeder, J Ast,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland*; M Döbeli,* ETH Zurich, Switzerland*; K von Allmen, A Neels, A Dommann,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland*; H Rudigier,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein, Switzerland*; B Widrig, **Jürgen Ramm**,* Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein

Typical coating systems in gas turbines are made of several layers, generally consisting of (1) a bond coat, which guarantee the mechanical stability over a wide range of temperature, (2) a thermally grown oxide, acting as an oxygen diffusion barrier and (3) a top porous ceramic, acting as a temperature barrier. Such a stack presents many interfaces where internal stresses and failure can occur. We will show here the capability of creating a complete layer stack for a bond coat by cathodic arc evaporation in an in-situ process sequence, i.e. without interruption of vacuum. Superalloy targets were produced from Ni-(Al-C-Co-Cr-Mo-Ta-Ti-W) and Ni-(Al-B-C-Co-Cr-Hf-Mo-Ta-Ti-W-Zr) powders by spark plasma sintering and processed in subsequent non-reactive and reactive modes. The phases, microstructure and composition of the powder, target and synthesized coatings are characterized by TEM, transmission EBSD, X-ray diffraction and RBS analyses and discussed. An epitaxial growth of the pure metallic coating can be observed on both multi and single crystalline superalloy substrates, which guaranty an enhanced mechanical stability of the first coating interface. The thin transition layer between the reactive and non-reactive depositions is composed of nano-crystalline partially oxidized Ni superalloy. Fully oxidized Ni superalloy and Al-Cr-O layers can be deposited on top. The thermal stability of the stack has been tested by in-situ high temperature XRD analyses as well as post mortem TEM, transmission EBSD and RBS analyses.

10:00am **A2-7 Steam Oxidation Behavior of $Yb_2Si_2O_7$-Based Environmental Barrier Coatings**, ***Kang Lee***, NASA Glenn Research Center, USA

Increased fuel efficiency is a game changer for gas turbines as fuel is the single most important cost, accounting for up to about 40% of the overall operation cost of commercial aircrafts. Increased fuel efficiency is obtained through increasing the thermal efficiency of the engine by increasing the overall pressure ratio (OPR). Increased OPR requires increased turbine inlet temperature, which is paced by advances in turbine hot section materials temperature capability. High turbine inlet temperature also contributes to environmentally friendly engines by reducing NOx. SiC/SiC Ceramic Matrix Composites (CMCs) are the most promising materials to revolutionize the temperature capability of turbine hot section materials because of their high temperature mechanical properties and light weight. Environmental barrier coatings (EBCs) is an enabling technology for CMCs by protecting CMCs from environmental degradation, especially from water vapor. One of the most likely EBC failure modes is oxidation by water vapor generated by combustion reactions (steam oxidation). The current state-of-the-art EBC is based on $Yb_2Si_2O_7$ and Si bond coat. In gas turbines water vapor migrates through $Yb_2Si_2O_7$ and oxidizes Si bond coat, forming a layer of silica (SiO_2), known as TGO, at the Si/$Yb_2Si_2O_7$ interface. As TGO grows in thickness with time the strain energy increases, ultimately leading to an EBC failure. This paper will discuss steam oxidation behavior of $Yb_2Si_2O_7$-based EBCs.

10:20am **A2-8 The Fatigue Behavior of TiCrAlTaSiN Coated and Uncoated Titanium Alloys**, *B Lerch*, ***Dongming Zhu***, *S Kalluri*, NASA Glenn Research Center, USA

Advanced multi-component TiCrAlTaSiN-based multilayered coatings were processed onto Ti-6Al-4V (Ti-6-4) and GammaMet PX (GMPX or TiAl) alloys by magnetron-enhanced physical vapor deposition technique. The coatings have been in developments aiming for improving advanced Ti alloy turbine engine component durability and oxidation-erosion resistance. In this work, the performance of the multi-component titanium nitride based coating systems (including the outer layer nitride based coating and inner TiCrAlSi barrier bond coat) was studied, and the coating influence on the fatigue behavior of the titanium-based alloys were compared. Although the multi-component coating was initially optimized to improve the coating high temperature oxidation and erosion resistance, the lower ductility nitride based coatings need further microstructure or composition optimizations to improve the mechanical stress resistance and stain tolerances for the highly loaded and high cycle fatigue turbine blade operating conditions, in particular for GMPX and other gamma TiAl alloys because the intermetallic Titanium alloys have limited ductility and are sensitive to defects. The preliminary results showed that the inner layer TiCrAlSi coatings had better adhesion and ductility, may help improve the oxidation resistance for the Ti alloys.

10:40am **A2-9 Crack Propagation Behavior of Thermal Barrier Coatings with Cyclic Thermal Fatigue Tests**, ***Dowon Song***, *T Song*, Hanyang University, Republic of Korea; *H Park*, *Y Jung*, Changwon National University, Republic of Korea; *J Zhang*, Indiana University Purdue University Indianapolis, USA

Crack formation and propagation behavior in thermal barrier coatings (TBCs) was observed through cyclic thermal fatigue (CTF) test for further understanding of TBC failure mechanisms. An analytical model was employed to predict residual stress distribution in the TBC samples to explain the experimentally observed crack-growth behavior. Initial cracks were formed on the surface and cross-section of TBCs prepared by air–plasma spray using Vickers indentation, and then CTF tests were performed for both cracked TBCs. Surface-cracked top coats were partially delaminated depending on their initial crack lengths that were formed with each loading level, suggesting a threshold surface crack length and showing a parabolic growth behavior. In the cross-sectional cracked TBC, cracks were strongly dependent on the direction, showing longer crack lengths in the direction parallel/horizontal to the interface between the top and bond coats than the direction perpendicular/vertical to the interface. The horizontal cracks grew in the similar manner as those on the surface, but the cracks in the vertical direction did not grow too much with CTF tests. All fatigue crack-growth rates show a negative dependence on stress intensity range in the Paris law due to the competing interaction of the residual stresses from indentation and thermal cycling. This study and analysis can be helpful for the further understanding of TBC failure in the design of reliable TBC systems.

Coatings for Use at High Temperatures
Room Royal Palm 1-3 - Session A3

Materials and Coatings for Solar Power Concentration Plants

Moderators: Vladislav Kolarik, Fraunhofer Institute for Chemical Technology ICT, Gustavo García-Martín, REP-Energy Solutions

2:30pm **A3-4 Corrosion Impact Of Alkali Carbonate At 750°C On Nickel Base, Stainless Steel And Alumina Forming Ferritic Steels**, ***Christine Geers,*** Chalmers University of Technology, Sweden

A carbonate mixture containing equal amounts of lithium, sodium and potassium was investigated in respect to its corrosive impact on metallic materials at 750°C to meet the demand of increased operation temperatures in concentrated solar power plants. A constant carbon dioxide feed was applied to suppress salt decomposition during exposure. Rapid oxidation and internal corrosion by carburization are characteristic consequences of carbonates in contact with steel at high temperatures. Significant differences in oxide scale stability and carbon ingress have been found for the three alloy classes after a maximum exposure time of 740 h. Depending on which oxide scale is formed, chromia or alumina, the corrosion features change from general to local occurrences pointing towards a high permeability of the oxide scale in the case of chromia formers compared to alumina formers where only locally non-protective behavior was observed. These observations are in agreement with findings from experiments in “solar salt” where major differences between alloys was also coupled to different oxide species and characteristics formed at the surface, independent of the permeating species which was nitrogen in that case.

2:50pm **A3-5 Challenges of New Materials and Coatings for Solar Receivers and Reflectors in Concentrated Solar Power Plants**, ***Florian Sutter,*** German Aerospace Center (DLR), Spain; *Y Binyamin,* Brightsource Industries, Israel; *A Agüero Bruna,* Instituto Nacional de Tecnica Aeroespacial (INTA), Spain; *C Hildebrandt,* Fraunhofer ISE, Germany; *D Fähsing,* DECHEMA Forschungsinstitut, Germany; *A Morales, A Fernandez-Garcia,* CIEMAT, Spain; *F Pérez-Trujillo,* Universidad Complutense de Madrid, Spain **INVITED**

With increasing share of fluctuating wind and photovoltaic power generation, Concentrating Solar Power (CSP) technologies with thermal storage become more important due to their flexibility in dispatching power to the grid. The International Energy Agency envisions that the global electricity share of CSP systems will reach 11% by 2050, provided that the CSP technology achieves significant cost reductions. One approach to meet this goal is the use of novel materials and coatings to increase the plant efficiency and to enhance lifetime.

This work reviews several new materials with potential to be applied in future CSP plants. In the low temperature range, protective coatings to prevent silver corrosion of glass mirrors will be discussed. In addition, experimental results of anti-soiling coatings to reduce the soiling rates on the mirror surface will be presented. In the medium temperature range (up to 400°C), novel selective absorber coatings operating in air or vacuum are described, as well as anti-reflective coatings with increased abrasion resistance. In the high temperature range (up to 750°C) a set of new high solar absorptance coatings for solar towers will be presented, as well as protective coatings for stainless steels to prevent corrosive attack from molten nitrate salts used as heat transfer and storage medium.

The above described materials are undergoing severe accelerated lifetime tests to ensure that they meet the challenging and harsh operating conditions, which materials need to face in CSP plants. The performance and expected lifetime of the prototype materials is compared to the state of the art.

3:30pm **A3-7 Corrosion Testing of Diffusion-coated Steel in Molten Salt for Concentrated Solar Power Plants**, ***Diana Fähsing,*** *T Meissner, M Galetz,* DECHEMA-Forschungsinstitut, Germany

In the course of energy transition the development of sustainable technologies for power generation providing base load supply is of particular importance. In comparison to photovoltaics Concentrated Solar Power (CSP) Systems have great potential to fulfil this requirement by the use of thermal storage systems employing molten salt mixtures as heat transfer fluids. For this purpose, molten nitrates are frequently discussed due to their beneficial thermal and physical properties as well as high working temperatures. These fluids are circulated through the superheaters of the CSP receiver for heat absorption, which is subsequently transferred to a steam generator for power generation.

While the receiver tubing's external surfaces are exposed to dust erosion as well as to fluctuating high temperature profiles, its insides are attacked by mechanisms from molten salt corrosion. In order to protect the piping system from degradation coatings can be applied to the materials in use which are commonly steels or Ni-based alloys. The goal is to achieve cost reduction to ensure an even more competitive position of the CSP technology with respect to other renewable sources on the market.

In this study, the behaviour of coated and uncoated ferritic-martensitic steels of type T91 and VM12 in molten salt (mixture of $NaNO_3$ and KNO_3) has been investigated under isothermal conditions. The diffusion coatings are based on potentially protective elements such as Al, Si or Cr and were applied to the steels either by pack cementation or slurry aluminization. Characterization of the samples was conducted by means of optical microscope, EPMA and XRD analysis in order to gain deeper understanding of the occurring corrosion mechanisms and for the purpose of life expectancy analysis.

3:50pm **A3-8 High Temperature Molten Salt Corrosion Behavior of Aluminide and Nickel-aluminide Coatings for Heat Storage in Concentrated Solar Power Plants**, ***Pauline Audigié,*** *S Rodríguez, M Gutiérrez,* Instituto Nacional de Técnica Aeroespacial (INTA), Spain; *V Encinas-Sánchez, F Pérez-Trujillo,* Complutense University of Madrid, Spain; *A Agüero Bruna,* Instituto Nacional de Técnica Aeroespacial (INTA), Spain

Thermal energy storage (TES) in concentrated solar power (CSP) plants is still a key issue as CSP plants are subjected to intermittency of the Sun. Using suitable TES systems is so of great interest. Currently, CSP plants use the so-called Solar Salt (60% $NaNO_3$ - 40% of KNO_3) as heat storage system which is considered to be stable, and has adequate heat storage and transfer capability. However, the maximum storage temperature is limited to 580ºC. New molten salt mixtures with higher temperature stability point are therefore required to increase the plants efficiency, all of this without increasing cost. In general, molten salts can be very corrosive to metallic components in direct contact with them and the corrosion resistance of the materials used for tanks or tubes depends on the formation of a protective oxide scale, which is similar to the protection mechanism occurring during oxidation in high temperature gaseous atmospheres. However, an important difference when using molten salts is that Cr, a protective oxide former, can be soluble in some salts mixtures preventing the formation of the oxide. This results in non-protective and/or fast-growing oxide formation and in the increment of material degradation due to higher corrosion rates. The use of coatings to prevent molten salt corrosion can be a solution. Slurry aluminide coatings are a low cost alternative that allows uniform coating of internal surfaces. Recent studies *[Dorched et al. 2016, Audigié et al. 2017]* have demonstrated the good behavior of these coatings but the protection mechanism is still not well identified. Moreover, since increasing nickel content in Ni-base alloys improved alloy corrosion resistance to molten nitrate-nitrite salt, newly developed nickel-aluminide coatings deposited on 9 wt.% Cr P91 were also tested in contact with molten salts. In this work, sprayed slurry aluminide and nickel-aluminide coatings deposited by means of electrodeposition and slurry application to 9 wt.% Cr P91 alloy have been characterized and both systems have been tested isothermally at 580ºC in contact with the Solar Salt and at 650ºC with a ternary molten salts mixture based on Na, K and Li carbonates. Both tests have been carried out under static and dynamic conditions. All the coated systems in contact with both salts up to 1000h performed much better than the uncoated material as they exhibited very slight weight variations and formed very thin Na or Li aluminates. On the contrary, the uncoated P91 developed a complex, fast growing multilayered oxide scales experiencing significant metal loss which was calculated after removing the corrosion products by chemical etching.

4:10pm **A3-9 High-Temperature Coatings for Protection of Steels in Contact with a Novel Molten Salt under Static and Flow-Accelerated Conditions for CSP Technology**, *V Encinas-Sánchez, M Lasanta, M de Miguel, G García-Martín,* ***Francisco Javier Pérez-Trujillo,*** Complutense University of Madrid, Spain

The dramatic increase in demand for energy independence and inclination towards renewable energy as a sustainable and green energy source have led research groups all over the world to concentrate their investigation on solar power. As renewable energy penetration grows, the need for utility-scale renewable generation with storage technology is increasingly important to mitigate intermittency problems, deliver power to peak demand periods and support transmission system reliability. Hence, concentrated solar power has gained momentum as an attractive technology. Molten nitrate salts are currently considered ideal candidates for heat transfer and storage applications because of their properties. However, this technology is still expensive compared to other renewable sources, which lead to propose solutions for reducing costs, such as development of new mixtures with greater work-temperature range. This increase in temperature limits would strongly require the use of alloys known for their high cost compared to other steels, such as ferritic-martensitic steels. According to this scenario, the use of high-temperature corrosion-resistant coatings would be a very suitable option, even more if they were deposited on ferritic-martensitic steels. This solution not only would help to overcome the corrosion problems of using ferritic-martensitic steels at higher temperatures, but also would allow the CSP industry to improve the Levelized Cost of Energy by reducing Operating & Maintenance costs. In this respect, alumina-based and zirconia-based sol-gel coatings seems to be a great option, both from an operational and economical point of view.

In this work, sol-gel alumina-based and zirconia-based coatings deposited on P91 has been tested in contact with a proposed novel molten nitrate salts at 560°C, results being compared with the uncoated substrate. The study was developed up to 1000 h under static and flow-accelerated conditions, the latter being performed in a novel pilot plant facility, patented under the reference code WO2016102719. Samples were characterized via gravimetric, SEM-EDX, and XRD. Also corrosion was monitored by electrochemical sensors, patented under the reference code WO2017046427.

Results showed the good behavior of the coated substrates, with very little weight gain after 1000 h of test in comparison with the uncoated ones, which exhibited significant weight gain and spallation. The good behavior of the proposed coatings was also observed by SEM-EDX and XRD. Furthermore, corrosion monitoring system showed the protective behavior of the coatings, these being compared with the uncoated samples, where widespread corrosion was determined.

Coatings for Use at High Temperatures
Room California - Session A1-2

Coatings to Resist High Temperature Oxidation, Corrosion, and Fouling

Moderators: Vladislav Kolarik, Fraunhofer Institute for Chemical Technology ICT, Shigenari Hayashi, Hokkaido University, Sebastien Dryepondt, Oak Ridge National Laboratory, USA

1:30pm **A1-2-1 Effect of Pre- and Post-Coat Processing on the Fatigue Life of Coated Disk Alloys**, ***James Nesbitt**, T Gabb, B Puleo,* NASA Glenn Research Center, USA*; R Miller,* Vantage Partners, USA **INVITED**

Increasing temperatures in aero gas turbines is resulting in oxidation and hot corrosion attack of turbine disks. Since disks are sensitive to low cycle fatigue (LCF), any environmental attack, and especially hot corrosion pitting, can seriously degrade the life of the disk. Application of metallic coatings is one means of protecting disk alloys from this environmental attack. However, since LCF is sensitive to surface conditions, pre- and post-coat processing can have a significant effect on the LCF life of coated bars.

Various pre-and post-coating conditions were examined with a Ni-45wt.% Cr coating applied by High-power impulse magnetron sputtering (HiPiMS) to a Ni-based disk alloy. Initial pre-coat surface conditions examined a grit blasted surface. Later pre-coat surface treatments involved wet blasting, two shot peening levels (8N-200% and 16N-200%) and a highly polished surface. Post-coat treatments involved two shot peening levels (4N-100% and 16N-200%) and a post-coat diffusion anneal in high purity Ar or a low PO_2 environment to encourage chromia scale formation. Half of the coated and uncoated bars were LCF tested at 760°C without further environmental exposure to evaluate the pre- and post-coat treatments. The second half of samples were either oxidized for 500 hours at 760°C in air, hot corroded for 50 hours at 760°C in air using a Na_2SO_4-$MgSO_4$ salt, or subjected to both exposures. Results of the LCF testing and post-test characterization of the various coatings will be presented and future research directions discussed.

2:10pm **A1-2-3 High-temperature Oxidation Resistance of Chromium-based Coatings Deposited by DLI-MOCVD for Enhanced Protection of the Inner Surface of Long Tubes**, ***Alexandre Michau**,* CEA, France*; F Maury,* CIRIMAT, France*; F Schuster, J Brachet, E Rouesne, M Le Saux,* CEA, France*; R Boichot, M Pons,* SIMaP, France

For nuclear safety issues, there is an international effort to develop innovative “Enhanced Accident Tolerant Fuels” (EATF) materials. EATF cladding tubes are of particular interest because these components constitute the first barrier against radioactive fission species dispersal in case of accidental scenario such as LOCA (Loss of Coolant Accident). One of the EATF development objectives is thus to improve drastically the high-temperature steam oxidation resistance of the cladding tubes in LOCA situation (temperature up to 1200°C, water steam environment…). Let us recall that the actual nuclear fuel cladding tubes are made from Zr-based alloys (1cm in diameter, 0.6mm thick and more than 4m long).

Several alternatives for outer wall protection have been proposed worldwide but there is currently no solution for the inner wall protection. Upon LOCA transients, the fuel cladding may experience ballooning and burst due to the inner pressure and the high temperature achieved. Then, its internal surface can be exposed to steam environment at high temperature, leading to fast inner oxidation and other secondary degradation effects, which induce significant embrittlement and degradation of the claddings.

In order to resist to this harsh environment, Cr-based coatings deposited by low pressure DLI-MOCVD (Direct Liquid Injection of MetalOrganic precursors) have been investigated. These hard coatings could also be of use in high-temperature oxidative and corrosive environment such as in aeronautics and other industries to protect components with 3D complex geometries.

The process has been optimized to ensure a thickness uniformity along the tube through experimental studies coupled with numerical modeling of the reaction mechanism and the whole deposition reactor. Thanks to a suitable chemistry of the Cr precursor, bis(ethylbenzene)chromium, different coatings were deposited including: metal Cr, chromium carbides Cr_xC_y and mixed carbides $Cr_xSi_zC_y$.

The high-temperature behavior of these Cr-based coatings under oxidative atmospheres has been studied: HT XRD, TGA and various oxidation tests including pure steam environment followed by water quenching at room temperature to be representative of LOCA situations. Amorphous Cr_xC_y coatings showed the most promising properties. The results are discussed in terms of oxidation mechanisms and protection of the fuel claddings inner surface deduced from fine characterizations of the samples before and after the tests.

2:30pm **A1-2-4 A New Process to Produce Localized Chrome Coating and Platinum-Modified Chrome Coating for Protection against Type II Hot Corrosion**, ***Zhihong Tang**, J McConnell, K Garing, S Sweeney,* Praxair Surface Technologies, Inc., USA

With increasing operating temperatures and more severe environments in today’s gas turbine engines, there is a need for a localized chromium diffusion coating for protection against type II hot corrosion and stress corrosion cracking. Conventional pack and vapor phase chromizing process have their limitations of applying such a local chrome coating. Masking for these processes is costly and often unsatisfactory. Therefore, pack and vapor phase chromizing process typically coat the entire part surface and then require undesirable post-coating treatment to remove excess chrome coating from locations where it is unwanted.

This presentation will discuss a new slurry based process that produces a localized chromium diffusion coating on the selected regions of gas turbine components in a controlled and accurate manner. No post-chromizing treatments such as machining or chemical treatment are required to remove excess chrome coating in comparison to conventional process. The resultant coating chemistry and microstructure meet the OEMs’ specifications. Other advantages of this new process include capability to coat large parts, and achieve uniform coating on parts having complex geometries such as shank and fir-tree root of turbine blades. This new slurry based process has been in production at Praxair Surface Technologies since 2015 yielding robust consistency in coating thickness and chemistry.

Based on this localized chrome coating technology, a platinum-modified chrome (PtCr) coating is under development. The addition of platinum can further enhance the resistance of chrome coating to type II hot corrosion attack. The PtCr coating process and its compatibility with other coating processes will be discussed.

2:50pm **A1-2-5 Characterization of Films Fabricated on AZ31 Magnesium Alloy by Heat Treatment and Immersion Methods**, ***Hyunju Jeong**,* Pohang Iron and Steel Company (POSCO), Republic of Korea

There has been considerable interest in the use of magnesium and magnesium alloys because of their outstanding properties, although their low corrosion resistance inhibits widespread applications. Therefore, many studies have been carried out to investigate the corrosion behavior of magnesium and magnesium alloys. Various coating techniques have also been proposed to improve their surface properties and corrosion resistance.

In the present work,thin films fabricated by immersion in solution, deionized water and NaOH solution were studied on AZ31 magnesium alloy subjected to heat treatment**.** The films were grown by active reactions at the interface of the magnesium alloy and the solution due to the high temperature of the substrate. Surface morphologies showed that the surface of the magnesium substrates was covered by dense films formed in the initial stages of immersion. The results revealed that the surface structures were different from those of films grown by simply immersing in aqueous solution. Cross-sectional results showed that the films formed in NaOH solution with heat treatment were denser than those grown by DI water immersion. X-ray photoelectron spectroscopy results indicated that the films fabricated by immersion in solution with heat treatment were mostly composed of MgO and $Mg(OH)_2$ in both the outer and inner layers. Potentiodynamic polarization measurements revealed that the thin films could improve the surface properties, acting as passive layers.

3:10pm **A1-2-6 Degradation Processes of LSM Based Interconnector Coatings under the Conditions of Pressurized Steam Electrolysis**, ***MariadelMar Juez Lorenzo**, V Kolarik, V Kuchenreuther-Hummel,* Fraunhofer ICT, Germany*; M Pötschke, D Schimanke,* Sunfire GmbH, Germany

To ensure energy for mobility in a post-fossil future, power-to-liquids converting electric power from renewable energy sources into liquid fuels is one of the approaches that have attracted attention. The steam electrolysis used to split water to hydrogen and oxygen is run under pressure up to 30 bar at temperatures around 850°C in order to achieve a high efficiency of the process. The impact of such severe conditions on the interconnector materials and coatings designed to retain the evaporating chromium is a crucial issue and needs detailed understanding to ensure a reliable operation.

Coatings of the type La-Sr-Mn oxide (LSM) were deposited on the interconnector material Crofer 22 APU by thermal spraying and by roll coating, the latter as a cost-efficient method. The samples were exposed in laboratory test autoclaves to 850°C and 30 bar in 100% water vapor up to 1000 h and in pure dry oxygen up to 3000 h, both representing the opposite extremes of the possible process atmospheres.

The LSM coating shows in water vapor a grain coarsening after 1000 h as well as a separation of grains with higher La content accumulated in the surface area and grains containing Mn in the interface area. Cr is detected very weakly in the areas with high La contents. In water vapor the oxide scales on the interconnector material with LSM coating are notably thinner than on the uncoated material. However, the LSM coating deposited by roll coating decomposes with the time to a coarse-grained lanthanum-rich phase in the surface area as well as in vicinity of the oxide scale and to a manganese-rich phase concentrated between them. The thermally sprayed LSM coating is more efficient in leading to thinner oxide scales, probably to its higher density. Additionally, it shows a less pronounced separation into the two phases.

After exposure to pure oxygen the LSM coating exhibits a homogeneous morphology with higher porosity in the case of the roll coating than for thermal spraying. Cr stemming from evaporation was found in the whole LSM coating when deposited by rolling and concentrated on the coating surface when thermally sprayed. The phases binding the Cr were identified by X-ray diffraction. After 1000 h the semi-conducting $LaCrO_4$ and $SrCrO_4$ are formed and after 3000 h $CrMn_2O_4$, which exhibits an electrical conductivity an order of magnitude higher than that of Cr_2O_3. The degradation processes influence the electrical conductivity of the chromium barrier coating.

3:30pm **A1-2-7 The Hot Corrosion Resistance of Hot-dip Aluminized Low Carbon Steel with Nickel Interlayer under Static Load***, Huan-Chang Liang,* *C Wang,* National Taiwan University of Science and Technology, Taiwan

The failure mechanism of hot-dip aluminized (HDA) low carbon steel with nickel interlayer under static loading was studied. The nickel interlayer was plated using electric method with a current density of 1.8 A/cm^2 for 10 minutes in Watts bath, followed by hot-dipped in molten pure aluminum (> 99.5 %) for 10 seconds at 700 °C. The specimens were smoothly deposited with 2 mg/cm^2 of $NaCl/Na_2SO_4$ salt mixture, with a portion of 50/50 wt. %. The tensile test with hot corrosion, induced by salt mixture, was carried out by applying a static load at 750 °C. The results show that the intermetallic layer of Ni/Al exhibits great adhesion and formability while the elongation of gauge length rising to 7 percent. Owing to the inter-diffusion between Ni/Al, the intermetallic layer gradually changes to laminar structure with higher nickel content which also contributes to enhance the performance. Meanwhile, hot corrosion induced by salt mixture reduces the amount of aluminum of the outer layer and lowers the thickness of Ni/Al intermetallic layer. As the perpendicular cracks formed, the salt mixture penetrate to the interface between substrate and coating layer causing the oxidation of the carbide and accelerating the phenomenon of break away. Overall, the HDA with nickel interlayer on low carbon steel could increase the lifetime about 5 times compared to bare material.

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 °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.

This work was supported by Institute of Materials Science of Silesian University of Technology, as a part of Statutory Research no BK-225/RM0/2017.

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 ºC 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 ºC 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 1500ºC 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.

Coatings for Use at High Temperatures
Room California - Session A1-3

Coatings to Resist High Temperature Oxidation, Corrosion, and Fouling

Moderators: Vladislav Kolarik, Fraunhofer Institute for Chemical Technology ICT, Shigenari Hayashi, Hokkaido University, Sebastien Dryepondt, Oak Ridge National Laboratory, USA

8:00am **A1-3-1 A Framework for Modelling the Nanomechanical and Nanotribological Properties of High Temperature HfB_xC_y Coatings**, ***Mohammad Humood**, T Ozkan,* Texas A&M University, USA*; E Mohimi, J Abelson,* University of Illinois at Urbana-Champaign, USA*; A Polycarpou,* Texas A&M University, USA

High aspect ratio conformal HfB_xC_y coatings were synthesized using low-temperature chemical vapor deposition (CVD). The carbon content was varied in the aggregates, which resulted in thin films with different compositions and mechanical properties. A framework was developed based on mixing of different aggregates to predict the nanomechanical properties of these films. Under this framework, we assumed the growth to be either diffusion or nucleation controlled. Different volume fractions of aggregates were considered. Certain mixing rations agreed well with the instrumented nanoindentation and provided further insights to better understand the results of and nanoscratch experiments. Nanoscratch experiments revealed the coefficient of friction (COF) to diminish to a superlubricity level of 0.05 when the carbon content increases in HfB_xC_y thin films. This value is comparable to DLC, and underlines the immense potential of HfB_xC_y hard thin film coatings for tribological applications. However, due to the shortcomings of C-based coatings such as a-C and DLC, which all experience graphitization and degradation under tribological contact conditions at elevated temperatures above 350 °C, there is a potential need for using ultra-high temperature ceramic coatings such as HfB_xC_y as an alternative for tribological applications.

8:20am **A1-3-2 Characterization of Thermal Properties of Different Pyrochlore Ceramic Materials Dedicated for Application as an Insulation Layers in Thermal Barrier Systems**, *M Mikuskiewicz, **Damian Migas**, G Moskal,* Silesian University of Technology, Poland

The basic direction of thermal barrier coatings (TBC) systems development is related to new materials solutions dedicated for bond-coats and ceramic insulating layers. From technological point of view this development consists mainly new conceptions in internal architecture of ceramic sublayer in the form e.g. of segmented, composite or multilayered morphology. In the case of new materials concepts the most interesting areas of investigations are related to strongly defected systems such as pyrochlore ceramic with overall formula RE2(Zr,Hf,Ce)2O7, perovskites or hexaaluminates materials of REZrO3 and (A,B)Al11O19 type respectively, or defected cluster zirconia based materials with two rare earth elements (with high cations ionic size differences) and formula ZrO2-Y2O3-RE12O3-RE22O3.

In presented article thermal properties of different types of pyrochlore material based on zirconia, hafnia and ceria of samarium are presented. Analyzed materials were synthesized by solid state reaction method form mixture of feedstock nano-sized powders of zirconia, hafnia, ceria and samaria. Synthesized materials were analyzed from its chemical and phase constituent point of view. The crystallite size was determined as well by X-ray diffraction method. Additionally the crystallite size and theirs orientations were analyzed by EBSD method. The morphological characterization of used feedstock powders was showed as well. The basic range of investigations was related to thermal parameters such as thermal diffusivity, specific heat and coefficient of thermal expansion analysis. Those data were obtained by laser flash analysis, calorimetric and dilatometric investigations respectively. On the base of those data the thermal conductivity was calculated in temperature range 25 to 1500°C. Obtained value of thermal parameters were compared to analogous data for usually used zirconia based ceramic of 8YSZ type.

The research has been supported by National Science Centre within Sonata scheme, under contract UMO-2016/21/D/ST8/01687.

8:40am **A1-3-3 Development of High Performance Corrosion Resistant Coatings using Graphene**, ***Anand Khanna**, K Aneja,* IIT Bombay, India

Graphene based high performance coatings have been developed using a graphene powder prepared in our lab using a new pressure based exfoliation method. Three kinds of coatings were made: (i) pre-treatment coatings on steel substrates using a new patented method of functionalizing graphene. The thin five micron coating has excellent adherence and very low permeability. (ii) graphene dispersed epoxy primer whose properties appear superior than a epoxy zinc rich coating or inorganic zinc rich coating and (iii) a graphene based polyurethane top coat with superior UV blocking properties. Combining all the individual coatings as conversion coating, primer and top coat, it becomes an excellent high performance coating with very high corrosion resistance, mechanical properties and weathering resistant. Each individual coating has its independent application for example pre-treated graphene can be an excellent replacement for electrolytic coating for automobile bodies and graphene dispersed epoxy can be a good replacement for epoxy based zinc rich coating or inorganic zinc rich primers.

9:00am **A1-3-4 Wide-range and Enhanced Filtration of Polyacrylonitrile Membrane for Water Purification by Coating with Thin Film Metallic Glass**, ***Shewaye Kassa**, Y Liao, J Chu, J Chen,* National Taiwan University of Science and Technology (NTUST), Taiwan

We have successfully fabricated comprehensive thin-film metallic glass (TFMG) coated polyacrylonitrile (PAN) membrane for wastewater purification. Several PAN-based membranes, synthesized via electrospinning were compacted into a single membrane through Zr-based TFMG coating by a means of magnetron sputtering deposition. TFMG coatings with various thicknesses ranging from 200 nm to 320 nm were grown on the membranes with no external heating. After coating with TFMG, the water purification performance of the membrane for synthetic wastewater, contaminated with toxic heavy metals (Cr and Cd), vegetable oil and microorganisms (E. coli and P. aeruginosa), was found to be higher than 95% with a pure water flux rate of 814 L $m^{-2}h^{-1}$. TFMG-coated PAN membrane exhibited extraordinary selectivity and a remarkable fouling recovery rate in comparison to the bare polyacrylonitrile membrane, which can be as a result of significant enhancement in the strength as well as the thermal and chemical stability of the membrane through the TFMGs coating.

9:20am **A1-3-5 The Effect of Surface Aluminizing to Enhance High-temperature Air-oxidation Resistance of Equimolar FeCoNi and FeCoNiCr Alloy**, ***Wu Kai**, F Cheng, F Chien, R Huang,* National Taiwan Ocean University, Taiwan*; J Kai,* National Tsing Hua University, Taiwan

The effect of surface aluminizing treatment on air-oxidation behavior of equimolar FeCoNi-based alloys (FeCoNi and FeCoNiCr) was studied at 950^{o}C. The results showed that the oxidation kinetics of the aluminized alloys followed the two-stage parabolic rate law. The optimal aluminized parameters were to heat the alloys at 850^{o}C for 4 h in 63% Al_2O_3-30% (Fe-Al)-7%AlF_3 powders under an Ar-gas flow-rate of 200 cm^3/min. The oxidation rates of the aluminized alloys were significantly lower than those of the untreated alloys by 3 to 5 orders of magnitude. The scales formed on the aluminized alloys consisted of an exclusive thin-layer of a-Al_2O_3 whose formation is responsible for the significant reduction of the oxidation rates with respect to the ternary and quaternary substrates.

9:40am **A1-3-6 TEM Study of Hf-B-Si-C-N Coatings Microstructure at High Temperatures**, ***Yi Shen**, M Zhang, J Jiang,* University of Texas at Arlington, USA*; J Vlček,* University of West Bohemia, Czech Republic*; E Meletis,* University of Texas at Arlington, USA

Amorphous $Hf_7B_{23}Si_{22}C_6N_{40}$, $Hf_7B_{23}Si_{17}C_4N_{45}$ and $Hf_6B_{21}Si_{19}C_4N_{47}$ coatings were synthesized by reactive pulsed dc magnetron co-sputtering. These coatings possess a hardness of ~ 20 GPa and a Young's modulus of ~170 GPa, and exhibit superior high-temperature oxidation resistance. The microstructures of the coatings annealed up to various temperatures from 1100 °C to 1600 °C in helium and in air were studied by X-ray diffraction (XRD) and transmission electron microscopy (TEM) to understand the effect of slight composition tuning on their microstructure evolution at high temperatures. All annealed films were found to have a two-layered structure composed of the original film followed by a nanocomposite oxidized surface layer involving HfO_2 nanoparticles embedded in a SiO_x-based matrix. Slight changes in the nitrogen content of the coatings were found to result in significant microstructure difference and oxidation resistance at high temperatures.

This work was supported by the U.S. NSF under Award NSF/CMMI DMREF-1335502.

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