Tuesday Afternoon, November 4, 2003

Advanced Surface Engineering
Room 323 - Session SE-TuA

High Temperature Protective Coatings
Moderator: A. Matthews, University of Hull, UK

2:00pm **SE-TuA1 Synthesis and High Temperature Performance of SiAlON Thin Film Coatings***, J.I. Krassikoff, G.P. Bernhardt, M. Call,* ***R.J. Lad,*** University of Maine

SiAlON ceramics made by alloying Al@sub 2@O@sub 3@ and Si@sub 3@N@sub 4@ possess oxidation resistance, high strength, and thermal shock resistance, which make them extremely attractive for high temperature coating applications. However, most work to date has emphasized bulk sintered SiAlONs rather than thin coatings. We have synthesized well-defined SiAlON thin films using rf magnetron sputtering of Al and Si targets in Ar / O@sub 2@ / N@sub 2@ mixtures on sapphire substrates. By manipulating the relative amounts of the deposition fluxes, homogenous versus multilayer or gradient SiAlON structures with a range of stoichiometries were produced. The films remain amorphous even with post-deposition annealing in vacuum or air up to 1000°C. Moreover, the film stoichiometries remain stable at high temperature in vacuum but lose nitrogen during air annealing. A thin film Ni-Cr corrosion sensor was embedded at the SiAlON / substrate interface, and the rate of oxygen penetration through the SiAlON film structures was measured in situ during accelerated thermal cycling tests in 1000°C oxidative environments. The time to failure ranges from hours to weeks depending on the exact SiAlON composition. Multilayer films with an Al@sub 2@O@sub 3@ overlayer exhibit the best oxidation resistance. Pin-on-disk wear tests indicate that the wear rate is also a function of the SiAlON stoichiometry. Our results yield important information that can be used to design and fabricate multifunctional SiAlON environmental barrier and thermal barrier coatings.@footnote 1@ @FootnoteText@@footnote 1@ Supported by AFOSR Grant #F49620-02-1-0323.

2:20pm **SE-TuA2 Synthesis and Characterization of Nanolayered TiO@sub x@/Al@sub 2@O@sub 3@ Coatings for Possible Elevated Temperature Applications***,* ***K.W. Lee****, M. Sturino, Y.W. Chung, L.M. Keer,* Northwestern University

It was demonstrated from previous studies that nanolayered coatings with the correct choice of components and layer thicknesses have enhanced hardness, due to interfaces providing barriers against dislocation motion and multiplication. We expect superlattice coatings made of two immiscible components to be stable against interdiffusion. Therefore, the layer structure and reasonable hardness for such nanolayered coatings should be preserved at high temperatures. These thermally stable coatings are desirable for protection of cutting tools in dry machining applications, which may operate at temperatures in the 800-1000@super o@C regime or higher. For this reason, TiO@sub 2@ and a-Al@sub 2@O@sub 3@ were chosen for this investigation. Nanolayered coatings made of these two immiscible components were synthesized by dc dual-cathode reactive magnetron sputtering. Substrate rotation was used to enhance uniformity of the coating. Substrates include M2 steel and Si(001). These coatings were exposed to air at 1000@super o@C. Coatings deposited on silicon were characterized before and after the heat treatment in terms of surface roughness, residual stress, and nanoindentation hardness. Actual dry machining will be performed to evaluate the performance of these coatings. These results will be presented and discussed in terms of the coating potential in dry machining and high-temperature tribological applications.

2:40pm **SE-TuA3 Advances in Surface Engineering for High Temperature and Wear Resistant Applications***,* ***A. Inspektor,*** Kennametal Inc. **INVITED**

Many components, when exposed to aggressive environments like high temperatures and cyclic stresses, fail when their surfaces have degraded beyond a predetermined limit. Thus, the demand for improved performance requires early integration of coatings into product design in order to assemble a functional surface tailored to resist the particular environment in which the component is working. This functional surface, with a unique combination of bulk and surface properties, is vital to the successful performance of the component. The corresponding surface engineering technique is an integral part of the manufacturing process. This paper will review present and future trends in surface engineering with an emphasis on the design and on the preparation of functional surfaces for high temperature and for wear resistant applications. Topics to be covered are nanostructured coatings architecture and substrate-surface integration for improved hardness, thermal stability, oxidation resistance and tribological properties of cutting tools and of wear parts. An effort will be made to identify performance needs in these applications and the corresponding surface engineering solutions. A discussion will be held how to meet the challenge of surface engineering and of substrate-coating integration in materials design.

3:20pm **SE-TuA5 Processing and Properties of NiAl-Hf Coatings via DC Magnetron Sputtering***,* ***B. Ning****, M.L. Weaver,* The University of Alabama

Crystalline NiAl-0.1Hf coatings (20 µm thick) were deposited onto CMSX-4 substrates at temperatures ranging from room temperature up to 300 °C using direct current (DC) magnetron sputtering. The microstructures of the coatings were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The as deposited coatings were single phase, and exhibit dense columnar Zone T microstructures. Column sizes were observed to increase from approximately 250 nm at room temperature to more than 2 µm in diameter at 250 °C. Annealing in argon at 1000ÂºC for 1h resulted in densification of the coatings and in increased adhesion between the coatings and the substrate. Microstructural and compositional changes of the coating system after isothermal oxidation were studied using SEM and energy dispersive spectroscopy (EDS) techniques. The mechanical properties of the coatings were studied via nanoindentation in the as deposited condition, after annealing, and following various oxidation heat treatments. The hardness and modulus of the coatings were observed to correlate with oxidation time. Extended oxidation resulted in decreased hardness due to grain growth and diffusion induced precipitation and phase transformations. The results are discussed relative to conventional CVD aluminide coatings.

4:20pm **SE-TuA8 Surface Alloying of Aluminum Films by Electron Beam Evaporation on Zirc-4 Substrates and Hydrothermal Crystal Growth in Sub-critical Condition***,* ***S.T. Park****, R.H. Baney,* University of Florida

Waterside corrosion of the Zircaloy cladding encasing the uranium oxide pellets is one of the primary factors limiting high "burn up" of nuclear fuel in pressurized water reactor (PWR) nuclear reactors. High "burn up" can significantly impact plant safety and economics. This research has been performed to develop ceramic coating corrosion protection system. Aluminum films were deposited on Zircaloy substrates by electron-beam evaporation and surface-alloyed by controlled oxidation at near the melting temperature of aluminum. Two different oxidation procedures were employed to make compositional gradient compound layers. These gradient compound layers can increase the corrosion resistance and minimize the interface defects like grain boundaries that can occur in multilayer coatings. The substrate surface roughness, aluminum film thickness, and air oxidation time and temperature were varied. The durability of films was tested through the use of an autoclave test in sub-critical condition that is the same condition as in actual PWR. The samples were then evaluated to determine the film condition. Deposited films were characterized for morphology and elemental composition using field emission scanning electron microscopy (FE SEM), energy dispersive x-ray analysis (EDX), and auger electron spectroscopy (AES). AES analysis of the oxidized coatings showed that gradient compositions were obtained as expected, with Al, Zr, and O content varying through the coating thickness. Glancing angle x-ray diffraction (GAXRD) analysis also showed that variety of intermetallic and oxide phases (such as Al@sub 3@Zr, Al@sub 2@Zr@sub 3@, Al@sub 2@O@sub 3@, ZrO@sub 2@ and Zr@sub 3@O) were formed in the coatings during processing. Hydrothermal growth of well-faceted particulates was observed after autoclave test. They were identified to be hydrothermal synthesized aluminum hydroxide, Boehmite by GAXRD and transmission electron microscopy (TEM).

4:40pm **SE-TuA9 Surface Studies of the High Temperature Corrosion of Stainless Steel by Oxygen Contolled Lead-Bismuth Eutectic***,* ***A.L. Johnson****, D. Parsons, J. Manzerova, D. Koury, B. Hosterman,* University of Nevada, Las Vegas*; D.L. Perry,* Lawrence Berkeley National Laboratory*; J.W. Farley,* University of Nevada, Las Vegas

There has been a resurgence of interest in the use of oxygen controlled Lead/Bismuth Eutectic (LBE) and similar high Z liquid metals as neutron spallation targets and coolants in advanced technology reactors. LBE was used in the Russian Alpha class nuclear submarines, with encouraging results. We have been investigating the corrosion of some western steels by oxygen controlled LBE in a Russian test loop using a number of techniques including SEM, EDAX, XPS and sputter depth profiling. We found for some mixtures of composition and surface preparation an order of magnitude lower oxidation and dissolution, associated with a change in morphology of the protective oxide layer. We shall discuss the effects of surface and near surface preparation and composition on this interesting oxidation/dissolution corrosion system.

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