Advanced Surface Engineering
Room 2007 - Session SE1-FrM

Coatings with Enhanced Thermal Stability & MAX Phases
Moderator: A.A. Voevodin, WPAFB

8:00am **SE1-FrM1 Coatings for Aero Engine Applications: Current Status and New Developments*, C. Leyens,*** Technical University of Brandeburg at Cottbus, Germany **INVITED**

Increasing demands placed on the efficiency and performance of aero engine gas turbines have resulted in ever higher process temperatures; this requires high thermal stability of the materials systems applied. Also, with increasing temperatures, the lifetime of components becomes strongly affected by oxidation and hot corrosion processes that eventually limit the useful service temperatures. Therefore, coating systems have been developed that effectively protect gas turbine hardware against environmental attack and at the same time provide thermal insulation of the underlying superalloy component. In the cooler part of the engines, titanium alloys are used which might be complemented with the application of intermetallic titanium aluminides in the future which have a higher temperature capability. To date, no coatings for these material are available, however, current research is looking for adequate solutions. The present paper will review state-of-the art science and technology of coating systems for high temperature applications in the turbine part of aero engines. Here, thermal barrier coating systems are applied consisting of a duplex layer system which is comprised of a metallic bond coating and a ceramic top coating that provides thermal insulation. The paper will address currently used coating technologies and will deal with a number of materials-related aspects such as thermal conductivity of the ceramic coating, thermal stability, lifetime and failure. Current research for future materials systems with improved performance will be highlighted. Moreover, coatings development for titanium alloys and titanium aluminides will be addressed shedding light on an emerging field of new coating systems. Major emphasis is placed on the thermal stability of the coatings, their interaction with the reactive Ti-containing substrate and their effect on the mechanical properties of the component.

8:40am **SE1-FrM3 Effect of a BN Interlayer on the Tensile Strength of NiAl Coated Sapphire Fibers*, D.E. Hajas,*** RWTH-Aachen University, Germany*; S. Kyrsta,* CemeCon AG, Germany*; J.M. Schneider,* RWTH-Aachen University, Germany

Intermetallic Matrix Composites (IMCs) can be fabricated by diffusion bonding of coated fiber bundles. Continuous single crystal @alpha@-Al@sub 2@O@sub 3@ fibers were coated with NiAl, h-BN and h-BN-NiAl using Chemical Vapor Deposition (CVD) for the h-BN interlayer and Physical Vapor Deposition (PVD) for the NiAl matrix material. The strength of the coated fibers was evaluated by tensile testing and compared to the strength of uncoated fibers. The effect of temperature during diffusion bonding on the fiber strength was studied. The influence of the coating on the fiber strength was evaluated by etching experiments. Fibers without h-BN interlayer cracked during heat treatment and testing could not be performed. This may be due to thermal stress induced fiber fracture during cooling. However, fibers coated with h-BN interlayer retained approximately 60% of their initial strength after the heat treatment. Two types of morphological features were identified which in combination with thermal stresses may be responsible for the strength degradation of annealed h-BN-NiAl coated fibers: Surface diffusion of the @alpha@-Al@sub 2@O@sub 3@ into cracks in the h-BN interlayer and reaction of the @alpha@-Al@sub 2@O@sub 3@ with impurities on the fiber surface.

9:00am **SE1-FrM4 Decomposition Process of Ti@sub 3@SiC@sub 2@(0001) Thin Films*, J. Emmerlich,*** *D. Music,* RWTH Aachen University, Germany*; H. Willman, P. Eklund, H. Högberg,* Linköping University, Sweden*; J.M. Schneider,* RWTH Aachen University, Germany*; L. Hultman,* Linköping University, Sweden

MAX phases (M: early transition metal; A: group-A element (13-15), X: C and/or N), such as, Ti@sub 3@SiC@sub 2@ exhibit metallic (e.g. well-conducting and ductile) and ceramic (e.g. oxidation resistance, high Young's modulus of ~340 GPa) properties. The main reason for the interesting set of properties is the nanolaminated crystal structure consisting of twinned Ti@sub 3@C@sub 2@ slabs interleaved with an atomic layer of Si acting as mirror plane weakly bonded to the Ti@sub 3@C@sub 2@ slabs. Epitaxial Ti@sub 3@SiC@sub 2@(0001) thin films were deposited on Al@sub 2@O@sub 3@(0001) substrates using DC magnetron sputtering. In our earlier investigations, these samples were vacuum-annealed from 800-1400°C with in-situ x-ray diffraction analysis. Between ~1000 and 1100°C the onset of decomposition was observed followed by the complete decomposition of the film after 25h annealing at 1200°C. Here, we present a 4-stage decomposition model based on the findings of transmission electron microscopy and supported by ab-initio calculations. The Si out-diffusion and evaporation initiated at the surface at 1100°C is followed by the O-uptake and SiO evaporation resulting in Ti@sub 3@C@sub 2@ relaxation, detwinning, and TiC@sub 0.67@ formation by C-redistribution with void formation. Initial differential scanning calorimetry measurements on as-deposited Ti@sub 3@SiC@sub 2@(0001) films show that the MAX phase is stable up to at least 1400°C for cases, where Si out-diffusion and O-uptake are prevented by means of Ti@sub 3@SiC@sub 2@(0001) films mechanically put face-to-face and heated in an Al@sub 2@O@sub 3@ crucible in Ar atmosphere. This further supports the validity of the presented model.

9:20am **SE1-FrM5 Synthesis, Structure and Properties of New Types of M-A-X Phase Films*, U. Jansson,*** Uppsala University, Sweden **INVITED**

New thin film materials in the M-A-X systems have recently been given a considerable attention. In these systems M represents a transition metal, A is a so-called A-element typically from group 13-15 (e.g. Si, Al, Ge) and X is either carbon or nitrogen. An interesting group of compounds in these systems are the nanolaminated MAX-phases with a general composition M@sub n+1@AX@sub n@ where n=1,2 or 3. They can be described as nanolaminates of metal carbides or metal nitrides separated by A-layers. The nanolaminated structure gives rise to special chemical and physical properties that suggest a potential use of these materials in different thin film applications such as low-friction materials, thermal protection coatings and chemically resistant coatings. Today, these compounds can be deposited as thin films using e.g. magnetron sputtering. Depending on the process parameters, however, also other types of thin film materials can be obtained. This includes other compounds such as perovskites and metastable solid solutions of metal carbides/nitrides with extremely high A-contents. Other examples are nanocomposites of carbides and nitrides in an A-containing matrix or other types of self-organizing microstructures. During thin film growth all these different types of materials and microstructures may be formed and a careful tuning of process parameters is required. This paper will discuss how phase composition and microstructure can be controlled in e.g the Ti-Si-C, Ti-Al-C and Ta-Al-C systems. The possibilities to design functional coatings for various applications will be discussed.

10:00am **SE1-FrM7 Growth and Characterization of Ti-Al-C Thin Films Deposited by dc Magnetron Sputtering from a Ti@sub 2@AlC Compound Target*, J. Frodelius,*** *P. Eklund, H. Högberg, L. Hultman,* Linköping University, Sweden

The ternary carbides and nitrides referred to as the MAX phases exhibit properties combining typical ceramic characteristics such as resistance to oxidation and thermal shock with electrical resistivity values comparable to those typically found in metals. These merits suggest many application areas for MAX phases both in the form of bulk material, such as heating elements and gas nozzles, as well as functional thin films in sensors and microelectronics. The synthesis of well-defined epitaxial thin films has progressed lately by the development of a magnetron sputtering process from elemental sources. This method is, however, less suited for growth under industrial conditions. In this study we present the synthesis of Ti-Al-C films on Al@sub 2@O@sub 3@ (0001) as well as Mo substrates, using dc magnetron sputtering from a Ti@sub 2@AlC target.@footnote 1@ The growth, structure and properties of Ti-Al-C films were investigated by XRD, SEM, TEM, EDS, nanoindentation and four-point-probe measurements. The films were synthesized for a wide range of substrate temperatures from ambient to 1000 °C as well as different substrate bias and target-to-substrate distances. The growth of Ti@sub 2@AlC films on Al@sub 2@O@sub 3@ (0001) substrates appear to be restricted to a narrow temperature regime around 700 °C. Below that, films consist of TiC:Al and for higher substrate temperatures, there is preferred growth of epitaxial TiC cubic phase. These films are depleted in Al, as revealed by EDS measurements. @FootnoteText@ @footnote 1@ MAXTHAL 211, courtesy of Kanthal AB.

10:20am **SE1-FrM8 Reactive dc Magnetron Sputtering of Ti-A-CN (A=Si, and Ge) MAX-Phase Thin Films***, **H. Högberg**, P. Eklund, J. Emmerlich, J. Frodelius,* Linköping University, Sweden*; O. Wilhelmsson, U. Jansson,* Uppsala University, Sweden*; L. Hultman,* Linköping University, Sweden

The M@subn+1@AX@subn@(n=1 to 3) phases exhibit a unique set of properties, described as being a combination of metallic and ceramic, reflected by the values reported for Ti@sub3@SiC@sub2@ on good oxidation and thermal shock resistance as well as high electrical conductivity. These attributes that stem from an anisotropic hexagonal crystal structure formed by early transition metals (M), group 14-16 elements (A) and carbon or nitrogen (X) are shared by the ~60 MAX phases. However, the extent is dependent on choice of material system and stoichiometry. The structure allows for freedom in substitution of elements of all the three constituents, opening possibilities to further tailor the properties of these materials by synthesis of quaternary and higher-order phases. In this study, we present results from thin film growth of the quaternary Ti-Si-C-N and Ti-Ge-C-N systems on Al@sub2@O@sub3@(0001) substrates at temperatures in the range 500 to 1000 @supero@C, using d.c. magnetron sputtering from elemental sources and growth in Ar/N@sub2@ plasmas. XRD shows a temperature-dependent growth behavior for epitaxial ternary Ti@sub3@SiC@sub2@ and Ti@sub2@GeC as seen by the possibility to synthesize single-phase films at 850 @supero@C, while lower temperatures result in growth of cubic phases. Higher temperatures promote surface segregation of the A-element as particularly pronounced in the Ti-Ge-C system resulting in growth of less Ge-rich stoichiometries. The addition of N@sub2@ to single-phase films in the Ti-Ge-C system results in growth of films of lower crystalline quality and thickness, and with nucleation of cubic phases as the flow of N@sub2@ is increased.

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