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Room Live - Session LI-WeM2
Advanced Characterization Techniques Live Session

Moderators: Prof. Dr. Diederik Depla, Ghent University, Belgium, Dr. Prabhakar Mohan, Solar Turbines, USA

11:00am **LI-WeM2-1 Welcome, Announcements & Sponsor Thank You's**, ***Prabhakar Mohan (Mohan_Prabhakar@solarturbines.com)***, Solar Turbines, Inc. , USA

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11:15am **LI-WeM2-2 Influence of the Microstructural Evolution of YSZ TBCs on their Thermal Insulation Potential**, ***Germain Boissonnet (germain.boissonnet@univ-lr.fr)***, *G. Bonnet, F. Pedraza,* Université de La Rochelle, France **INVITED**

Keywords Thermal Barrier Coatings (TBCs), Thermal Diffusivity, CMAS, Oxidation

Abstract. In aeronautical gas turbine engines, the metallic materials employed in the hottest sections are subject to very harsh chemical environments at high pressures and temperatures. Thermal barrier coating (TBC) systems (ceramic yttria-stabilized zirconia (YSZ) / MCrAl or NiPtAl bond coatings / inner cooling system) are employed to lower the temperature at the surface of the components, which ensures an adequate thermomechanical behaviour and reduces the oxidation/corrosion rates. However, the increase of the turbine inlet temperature for enhanced engine performance brings about new degradation phenomena (e.g. CMAS) and loss of efficiency of the TBCs [1-4]. Therefore, understanding the evolution of the insulation ability of TBCs in such harsh environments is key from both the scientific and technological perspectives to estimate the lifetime of these coatings, hence that of the engines.

Based on current plasma-sprayed (PS) and electron-beam physical vapour deposited (EB-PVD) YSZ coatings, this work seeks to provide a better comprehension on the relationships between the intrinsic properties of the current TBCs and their thermal insulation capacity as a basis for the development of future coatings. Thermal ageing, in the presence or absence of CMAS, was performed on both type of coatings and showed that the sintering of the YSZ, the evolution of crystal phases, the reactions between YSZ and CMAS and the growth of thermal oxides alter the thermal diffusivity to different extents.

References

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11:45am **LI-WeM2-4 High-Entropy Ceramic Thin Films; A Case Study of Nitrides, Oxides and Diborides**, ***Paul Heinz Mayrhofer (paul.mayrhofer@tuwien.ac.at)***, *A. Kirnbauer, R. Hahn,* TU Wien, Institute of Materials Science and Technology, Austria; *P. Polcik,* Plansee Composite Materials GmbH, Germany **INVITED**

High-entropy materials often outperform their lower-entropy relatives in various aspects, such as thermal stability and fracture toughness. While there are extensive research activities in the field of high-entropy alloys, comparably little is performed for high-entropy ceramics. Here we show, that especially with physical vapor deposition the development of single-phased high-entropy ceramics is straight-forward. Or, are we just lucky? On the definition-basis for high entropy alloys, we use the term “high-entropy” for our nitrides, oxides and borides if at least five corresponding binaries constitute them, and the configurational entropy (per formula unit) amounts to at least 1.5R.

All high-entropy ceramic thin films investigated, outperform their commonly-used binary or ternary constituents in thermal stability and thermomechanical properties.

High-entropy nitrides, sputtered from equimolar powder-metallurgically-prepared targets, are single-phase fcc-structured with a hardness H comparable to those of the constituting binaries and ternaries, but considerably lower indentation moduli E. For example, H = 33 and 31 GPa with E = 450 and 433 GPa for (Hf,Ta,Ti,V,Zr)N and (Al,Ta,Ti,V,Zr)N; while H = 36 GPa with E = 520 GPa for (Ti,Zr)N. But even after vacuum-annealing at 1300 °C, the (Hf,Ta,Ti,V,Zr)N still showed 28 GPa of hardness and no clustering of atoms or indications for decomposition processes (based on atom probe tomography APT and XRD studies). Alloying with ~5 at% Si substantially increased their oxidation and failure resistance.

High-entropy (Al,Cr,Nb,Ta,Ti)-oxides always crystallized in single-phase rutile structure independent on the O_2-to-Ar flow-rate-ratio used (0.4–4; p = 0.4 Pa) during sputtering a metallic equimolar target. Thereby, simply R decreased from 33 to 20 nm/min, H increased from 22 to 24 GPa and E increased from 380 to 410 GPa. Vacuum annealing at 1200 °C solely led to a change of their nearly random crystal orientation towards a highly 101-texture.

The hardness of our as-deposited high-entropy $(Hf,Ti,Ta,V,Zr)B_2$ and $(Hf,Ta,V,W,Zr)B_2$ diborides (non-reactively sputtered from corresponding targets) is very high with 47 and 46 GPa, combined with E of 550 and 610 GPa. Even after vacuum-annealing at 1300 °C, the still single-phased $(Hf,Ta,V,W,Zr)B_2$ exhibits 45 GPa hardness and no indications for recovery and decomposition. Contrary, the ternary $(Ti,Zr)B_2$ already “softened” to 40 GPa upon annealing at 1100 °C.

These results confirm the beneficial effects of high-entropy also for ceramics, especially with respect to the three core-effects, severe lattice distortion, sluggish diffusion, and formation of single-phased crystalline solids.

12:15pm **LI-WeM2-6 Characterization of Defects and their Dynamics using Transmission Scanning Electron Microscopy**, ***Daniel Gianola (gianola@engr.ucsb.edu)***, University of California Santa Barbara, USA **INVITED**

The past several years has witnessed a surging popularity of scanning transmission electron microscopy (STEM) for defect characterization using diffraction contrast imaging. Advantages of these methods over conventional TEM include the suppression of dynamical effects and spurious contrast, as well as the ability to image relatively thick specimens. In parallel, the use of transmission modalities in the scanning electron microscope (SEM) has attracted recent attention.

Here, we link these capabilities by employing an field emission SEM equipped with an annularly-segmented STEM detector for defect characterization – termed transmission SEM (TSEM). Stacking faults and dislocations have been characterized in commercially pure aluminum, strontium titanate, a polycrystalline nickel-base superalloy, several multi-principal-element alloys, and a single crystal cobalt-base material. Imaging modes that are similar to conventional CTEM bright field (BF) and dark field (DF) and STEM are explored, and some of the differences due to the varying accelerating voltages highlighted. Defect images have been simulated for the TSEM configuration using a scattering matrix formulation, and diffraction contrast in the SEM is discussed in comparison to TEM. Interference effects associated with conventional TEM, such as thickness fringes and bending contours, are significantly reduced in TSEM by using a convergent probe (similar to a STEM imaging modality) enabling individual defects to be imaged clearly even in high dislocation density regions.

We further show that TSEM provides significant advantages for high throughput and dynamic *in situ* characterization. We employ location-specific *in situ* tensile experiments to study the nature of dislocations dynamics in several structural alloys. By selecting specific crystallographic orientations relative to the tensile axis, we observe the underpinnings of several plasticity mechanisms including shear localization, cross-slip, and dislocation junction formation and evolution. To demonstrate the power of this new method for defect-contrast studies, we further show the ability to deduce reciprocal space mapping, and thereby, Burgers vector determination.

12:45pm **LI-WeM2-8 Closing Remarks and Thank You's**, ***Diederik Depla (Diederik.Depla@ugent.be)***, Ghent University, USA

We hope you enjoyed the Live Session. Please join us for our Post-Session discussion and additional Q&A opportunities. We hope to see you tomorrow!

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