Advanced Characterization Techniques for Coatings, Thin Films, and Small Volumes
Room Pacific Salon 1 - Session H1-1-TuM

Spatially-resolved and In-Situ Characterization of Thin Films and Engineered Surfaces I

Moderators: Grégory Abadias, Institut Pprime - CNRS - ENSMA - Université de Poitiers, **Xavier Maeder**, Empa, Swiss Federal Laboratories for Materials Science and Technology, **Michael Tkadletz**, Montanuniversität Leoben

8:20am **H1-1-TuM-2 Evolution of the Nanoporous Structure of Sintered Ag Joints at High Temperature using In-Situ X-ray Nanotomography***, **Xavier Milhet**, A Nait-Ali, D Tandiang, L Signor,* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France*; M Legros,* Cemes - Cnrs, France*; Y Liu, D Van Campen,* Stanford Synchrotron Radiation Lightsource - SLAC National Accelerator Laboratory, USA

Silver pastes sintering is a potential candidate for die bonding in power electronic modules. The thin Ag joints, obtained by sintering, exhibit a significant pore fraction thus reducing the density of the material compared to bulk silver. This was shown to alter drastically the mechanical properties (Young's modulus, yield strength and ultimate tensile stress) at room temperature. However, while careful analysis of the nanoporous structure has been reported in 2D, little is known about its quantitative spatial evolution during thermal aging and more specifically during temperature jumps. In this context, high temperature evolutions of the 3D nanoporous structure were observed in-situ using a heater fitted into the beamline 6-2C of SSRL. Segmentation of the porosity and subsequent statistical analysis of the tomographic dataset reveal pore shape, size and spatial distributions evolution during continuous heating. Such an analysis provides insight into the microstructural evolution of sintered nanoporous Ag joints in-service.

8:40am **H1-1-TuM-3 Atom Probe Tomography to Help Understand Deformation Mechanisms in Metallic Alloys***, **Baptiste Gault**,* Max-Planck Institute for Iron Research, Düsseldorf, Germany*; P Kontis, S Makineni, J He, Z Peng,* Max-Planck Institut für Eisenforschung, Germany*; S Neumeier,* Friedrich Alexander-University Erlangen-Nürnberg (FAU), Germany*; J Cormier,* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France*; D Raabe,* Max-Planck Institut für Eisenforschung, Germany **INVITED**

Atom probe tomography (APT) is a burgeoning materials characterization technique that enables elemental mapping in three-dimensions at the nanoscale and with high elemental sensitivity. APT exploits the effect of an intense electrostatic field to cause the departure of individual atoms, in the form of ions, from the end of a very sharp needle-specimen. The very particular geometry of the specimen gives rise to a highly magnified image formed by the projected ions that are accelerated away from the specimen's surface by the electric field itself. This projection microscope is then coupled with a time-of-flight mass spectrometer to reveal the elemental identity of each of the detected ions. In this presentation, I will cover some of the basics of the technique, I will be showcasing some applications to investigate segregation phenomena induced by the plastic deformation of high-temperature alloys.

9:20am **H1-1-TuM-5 On the Chemical Composition of TiAlN Thin Films - Comparison of Ion Beam Analysis and Laser-assisted Atom Probe Tomography with Varying Laser Pulse Energy***, **Marcus Hans**, J Schneider,* RWTH Aachen University, Germany

We compare the chemical composition of TiAlN thin films determined by ion beam analysis and laser-assisted atom probe tomography (APT). The laser pulse energy during APT was increased subsequently from 10 to 20, 30, 40, 50, 100 and 200 pJ within a single measurement, covering the range that is typically employed for the analysis of transition metal nitrides. The laser pulse energy-dependent Ti, Al and N concentrations were compared to ion beam analysis data, combining Rutherford backscattering spectrometry and elastic recoil detection analysis with the total measurement uncertainty of 2.5% relative deviation. It can be learned that the absolute N concentration from APT is underestimated by at least 9% and the absolute Al concentration from APT is overestimated by at least 16%, while absolute Ti concentration values are for both techniques in good agreement. The here presented comparative analysis clearly shows that absolute Al and N concentration values obtained by ion beam analysis deviate significantly to the APT data for the laser pulse energy range from 10 to 200 pJ.

9:40am **H1-1-TuM-6 Microstructure and Oxidation States of Ni in Sub-Nanometric Layer Depending on its Seed-Layer (Zinc Oxide, Silver Layers): A Multi-Techniques Approach to Trespass Limits of Resolution***, **Justine Voronkoff**, H Montigaud,* Saint-Gobain Recherche/CNRS, France*; L Largeau,* CNRS/C2N, France*; S Grachev,* Saint-Gobain Recherche/CNRS, France

Functional glazing for thermal isolation consist of stack of layers deposited by PVD magnetron sputtering on flat glass substrates at room temperature. They combine metallic and dielectric thin layers in order to optimize reflectance in the IRs and transmission in the visible. The determination of layers microstructures and chemical states is of great interest for industrials to understand the macroscopic (optical, mechanical) properties of such stacks. Literature is restricted and suffers a lack of adapted techniques. Hence, this is of major interest to develop tools to understand those stacks microstructures.

For this study, we have focused our works on the behavior of ultra-thin layers of nickel or nichrome (< 1 nm) deposited on silver (10-20nm) and zinc oxide layers (5-20nm), which could be found in such functional stacks. First part is dedicated to investigations on the seed layer (ZnO and Ag) which are polycristalline. AFM images and GIXRD in coplanar and non-coplanar modest to reach key parameters such as crystal size, density of grain boundaries and surface morphology which drastically influences the NiCr layer characteristics. In addition to the heterogeous environment and the small thicknesses, the oxidation state of Ni and Cr itself impact the layer morphology, which make it challenging to characterize. In order to characterize the NiCr layer depending on the sputtering deposition conditions and its under-layers as best as possible, we use a multi-approach using combined high resolution techniques: AFM, GIXRD, XPS in situ, TEM and STEM-HAADF, Atom Probe Tomography. We use a XPS directly connected to the deposition chamber that enables following the growth at the first stages of the NiCr layer along the deposition process. Using a tilted XPS mode could give us information regarding the growth of NiCr (island or homogeneous layer) without any contact to the atmosphere. Moreover, XPS analysis gives access to the oxidation states of the different species in presence (Ni, Cr and the topmost part of the seed-layer). Coupled with ex situ STEM-HAADF it permits a 2D characterization of crystallites size and distributions, Ni coverage and microstructure depending on the different substrates (Ag, ZnO) and to have chemical information. ATP first results complete those revealed by STEM with the 3D distribution of the species with a sensibility much higher than EDS. All those techniques give complementary information with more or less advantages at different resolutions, which would be discuss and justify their combined use. Deposition conditions for the sputtered layers will be compared as they directly determine the properties discussed above.

10:00am **H1-1-TuM-7 Nanomechanical Investigation on Lateral fcc-w Phase Fields of a Partially Decomposed and Transformed Nano–lamellar CVD fcc–$Ti_{0.2}Al_{0.8}N$ Coating***, **Michael Tkadletz**, A Lechner, N Schalk,* Montanuniversität Leoben, Austria*; B Sartory,* Materials Center Leoben Forschung GmbH (MCL), Austria*; C Mitterer,* Montanuniversität Leoben, Austria*; C Czettl,* CERATIZIT Austria GmbH, Austria

In metastable, nano-lamellar chemical vapor deposited (CVD) fcc-$Ti_{1-x}Al_xN$ coatings, at elevated temperatures, intact fcc-TiAlN areas co-existing with non-lamellar fully decomposed and transformed fcc-Ti(Al)N and w-Al(Ti)N areas could be observed. It is assumed that the observed phase fields and their microstructure strongly correlate with their mechanical properties. To study this correlation, this work focuses on the investigation of a nano–lamellar CVD fcc–$Ti_{0.2}Al_{0.8}N$ coating in an intermediate sample state annealed at 1050 °C for 5 min, exhibiting fcc- and w-phase fractions side by side. A cross-section of the coating was characterized by means of scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD) measurements and subsequently used for nanomechanical testing. Modulus mappings were performed by evaluating the elastic response after superimposing a dynamically oscillating load to the contact force applied during scanning probe microscopy imaging. Arrays of low load quasistatic indentations on the respective positions provided the basis to create maps of the lateral hardness distribution with a resolution of ~100-200 nm. A cross-correlation of the results with SEM images and EBSD inverse pole figure maps allowed to clearly identify the lateral phase fields and their effects on the mechanical properties. Obtained moduli and hardness values were in good agreement with values measured on the as-deposited and fully decomposed/transformed state. The results of this study clearly demonstrate the power of correlative characterization techniques for the investigation of advanced hard coating materials at the nanoscale.

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