Advanced Characterization Techniques for Coatings, Thin Films, and Small Volumes
Room Grand Hall - Session HP-ThP

Advanced Characterization Techniques for Coatings, Thin Films, and Small Volumes (Symposium H) Poster Session

HP-ThP-1 Cyclic Tensile Deformation of Freestanding, Nanocrystalline NiTi Films using MEMS Stages*, **Paul Rasmussen**, R Sarkar, J Rajagopalan,* Arizona State University, USA

Controlling the micro/nanostructure of thin films would enable us to explicitly tailor their mechanical behavior. Here, a new process is described in which thin films can be synthesized with precise microstructural control via systematic deposition of nanometer-sized seed crystals, and subsequent crystallization of amorphous precursor films. Using this process, austenitic NiTi (nitinol) films with submicron grain sizes are synthesized. We then co-fabricated freestanding samples of the films with MEMS testing stages and performed cyclic tensile load-unload experiments. Chromium seeded samples showed a high phase transformation stress (> 700 MPa) during the first cycle, with a further increase in transformation stress during subsequent cycles. Unlike the pseudo-elastic behavior typically observed in microcrystalline nitinol, the film showed a continuous decrease in stress-strain slope during unloading. Preliminary in-situ TEM straining studies suggest that this unusual loading behavior is caused by a combination of reverse phase transformation and reverse plasticity.

HP-ThP-3 Ion Irradiation Behavior of a Nanocrystalline BCC High-Entropy Alloy*, Y Xiao, H Ma, A Sologubenko, R Spolenak, **Jeffrey M. Wheeler**,* ETH Zürich, Switzerland

Refractory high-entropy alloys (HEAs) have attracted significant attention due to their superior mechanical properties at elevated temperature, making them potential candidates for structural nuclear materials. However, there is little known about their radiation resistance, particular at harsh environment relevant for fission and fusion applications. Here, strongly textured, columnar and nanometer-size-grain NbMoTaW HEA thin films with and without ion beam assisted deposition (IBAD) are produced. They are irradiated with 4.5 MeV Au ions at 77 K and room temperature with doses up to 1000 displacement per atom at ion-channeling direction. Electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM) are used to characterize the microstructural changes. The examined HEA thin film with IBAD technique exhibits superior microstructural radiation resistance compared to normal deposited HEA and W ones. The radiation resistance can be correlated to mechanical properties via nanoindentation.

HP-ThP-4 Evaluation of Properties in Steel with Hard Coating under Hydrogen*, **Noe Lopez Perrusquia**,* Universidad Politecnica Del Valle De México, México*; M Doñu Ruiz,* Universidad Politecnica del Valle de México, México*; C Torres San Miguel,* Sección de Estudios de Posgrado e Investigación de la Escuela Superior de Ingeniería Mecánica y Eléctrica Unidad Zacatenco, Mexico*; V Cortés Suárez, J Garcia Sanchez,* Universidad Autonoma Metropolitana Azcapotzalco, Mexico*; L Sánchez Fuentes,* Universidad Politecnica del Valle de México, Mexico

In this study, on ASTM A36 steel surface with hardened, at 950 °C for 3, 5 and 7h; through dehydrated paste-pack boriding process. Then, They were investigated, the behavior of the specimen hardened superficially in the microstructure, the hardness, the present XRD phases and characteristics by three point bending. Simultaneously, was investigated the hydrogen permeation effect on the coating formed in the surface of the material and the mechanical characteristics, were evaluated by three point bending and hardness. Obtained a layer sawn with the time and temperatures study; likewise the growth of FeB/Fe_2B layers. There is a hardness change of the boron coating subjected to hydrogen permeation and without hydrogen permeation for each time and temperature. The three-point test showed changes in properties with the coating formed on the surface of the study material subjected to hydrogen permeation and without hydrogen permeation. Showing that the coating boron an efficient alternative to lessen the effect by hydrogen permeation.

HP-ThP-6 Coatings and Interfaces Characterization: Depth Profiling from the First Nanometer down to the Substrate using RF GD-OES*, **Philippe Hunault**,* HORIBA Instruments, USA*; M Chausseau, K Savadkouei,* HORIBA Scientific, USA*; P Chapon, S Gaiaschi,* HORIBA Scientific, France

With its capability to perform depth profiling on conductive and non-conductive materials with a nanometric resolution and to go up to 150 µm deep into the sample within few minutes, GD-OES is an ideal tool to evaluate depth profiles on materials and to study interfaces between layers, diffusion processes or to optimize coatings processes. Many elements can be analyzed simultaneously, including Oxygen, Hydrogen, Deuterium, Carbon, Fluorine, Sulfur, Lithium… GD-OES is a versatile tool to study materials that complements other techniques such as XPS and SIMS. Since recently, GD-OES can also be used for the measurement of layer thickness and odd shape samples can be characterized.

Results obtained on various nm thin and thick coatings will be shown during this presentation: The use of RF GD-OES for the optimization of electroplating processes will be described with depth profiles of coatings on both inorganic and organic substrates and the direct determination of thickness using Differential Interferometry. Some results obtained on non-conductive organic coatings used for Aluminum packaging will be shown as well as how GD-OES can be used for thickness measurement on Zinc coatings with a comparison with cross-sectional SEM data. Other examples will include the use of GD to manage quality issues such as unexpected elements at the coating/steel interface or for hot-rolled pipe production.

HP-ThP-7 *In situ* Measurement Setup for DC Magnetron Sputtering Thin Film Deposition*, **Quentin Herault**, S Grachev, I Gozhyk, H Montigaud,* Saint-Gobain Recherche/CNRS, France*; R Lazzari,* Institut des Nano Sciences de Paris - Sorbonne Université, France

DC magnetron sputtering is a common technique of deposition at the industrial scale. It involves complex phenomenon due to the variety of species involved, such as electron, ions, neutral, etc. Consequently, deposition parameters are the key to improve thin film quality. Among them, sample holder potential, deposition speed, deposition pressure, target-sample distance are generally identified as the most pertinent.

To understand the effect of these parameters, we developed different *in situ* measurements methods in the same chamber used during thin film deposition. Surface temperature, mechanical stress, optical reflectivity and resistivity measurement were chosen as complementary methods. Our *in situ* results, correlated to thin film morphology measured by *ex situ* measurements gave a good overview of the impact of deposition parameters on grain size and deposition steps for example.

We propose here to describe this setup and results obtained in the particular case of silver thin film deposition.

HP-ThP-8 Preparation and Physical Properties of Multiferroic $CaMn_7O_{12}$ Thin Films*, **Yu-Chin Tseng**,* National Chiao Tung University, Taiwan*; S Jian,* I-Shou University, Taiwan*; C Lin,* National Tsing Hua University, Taiwan*; J Juang,* National Chiao Tung University, Taiwan

This study is aiming at how to prepare epitaxial $CaMn_7O_{12}$ (CMO) films on various substrates by pulsed laser deposition (PLD). The growth temperature for preparing CMO thin films can be between 600 and 700℃. We have tried a wide range of deposition parameters, for example, varying the oxygen pressure from 0.1 to 8×10^{-5} torr and also tried to change the laser energy. When using the parameters of 8×10^{-2} torr at the 700℃ can successfully grow the CMO on the STO(100) substrates. The XRD results reveal that all of the diffraction peaks can be indexed to belong the CMO phase with no obvious preferred orientation and we further used synchrotron radiation sources for XRD measurements to confirm that the obtained samples are indeed with correct phases. In addition, we used phi scan technique to check the symmetry of our thin films. It is could obtain both the CMO thin film and the STO(100) substrate are displaying very clean 4-fold symmetry and displaces 45° with respect to each other. It is also interesting to note that, although the laser energy appeared to have little effects on the formation of crystalline phase and orientation, the AFM examination does reveal significant differences in the film surface morphologies. It is evident that with smaller laser energy, the grain structure appears to be more square-like and when the laser energy was increased, the film grain morphology became more spherical. The typical magnetic properties of the obtained CMO thin films are shown that the present CMO thin films exhibit two magnetic transitions at ~96K (T_{N1}) and ~42 K(T_{N2}), respectively that measured by SQUID, which in good agreement with the two antiferromagnetic transitions occurring at T_{N1}~90 K and T_{N2}~48 K reported previously. The fact that our CMO films shows higher T_{N1}

and lower T_{N2} is also interesting. Nevertheless, the reason that is most likely to cause this phenomenon at present is that the epitaxial strain introduced during film growth.

*) To whom correspondence should be addressed. Electronic mail: yctseng101@gmail.com

**HP-ThP-9 SIO X-Ray: View Inside your Material with Contact Experiments*,* *Nick Bierwisch, N Schwarzer,* SIO, Germany

Caused by the increasing complexity of materials, be it as coatings, multi-layers, fiber or particle reinforced structures or other forms of compounds, classical engineering methods, linear material models and rules of thumb aren't enough anymore.

Proper characterization and optimization of such structures requires invertible mathematical tools of sufficient holistic character. Therefore, SIO developed analytical models which can dramatically speed up the analysis and simulation of complex contact situations compared to FEM, BEM or other numerical systems.

Together with sophisticated measurement devices it's now possible to characterize even extremely complex material structures in a still completely generic (physical) manner.

The knowledge gained thereby, almost literary allows a view inside the material without cutting it open. In fact, our noninvasive (partially even non-destructive) X-ray technology, which is to say the combination of suitable material test with sophisticated physical analysis, allows a stunning presentation of the material interior with all field components, be it stresses, strains, energies etc.

This way a much easier and - what is more - almost entertaining method of finding initial failure mechanisms and detect weak material spots came into existence.

**HP-ThP-11 Glow Discharge Optical Emission Spectroscopy: Advances toward Quantitative Coating Compositional Depth Profiling*,* *Amir Tavakoli, F Li,* Air Liquide - Balazs NanoAnalysis Laboratory, USA

As of key analytical data for understanding of functional coatings behavior are (i) coating compositional depth profile, (ii) potential chemical interaction of the coating material with the substrate, and (iii) possible impact of processing environment on the coating composition. Glow discharge optical emission spectroscopy (GDOES) is known as a fast elemental analysis technique for qualitative compositional depth profiling and capable of detecting almost all elements at ppm level. Balazs NanoAnalysis Laboratory with many years of R&D efforts on the GDOES methodology development has proven records of semi-quantitative compositional depth profiling for a variety of metal and ceramic coatings in a rage of thickness from 10 nm to 50 µm. In this presentation, a systematic GDOES research work on a series of widely used coatings on aluminum alloys with emphasis on anodized coatings is reported. It is shown how major, minor, and trace element concentrations change from the top-coating surface to the substrate, how the surface elemental composition can be affected from the processing conditions, and how the coating composition is influenced by the diffusion of the substrate constituent elements (coating-substrate interdiffusion). In addition, the reliability of the elemental quantification by GDOES measurements are cross checked using the other elemental analysis techniques.

Author Index

Bold page numbers indicate presenter