Advanced Characterization Techniques for Coatings, Thin Films, and Small Volumes
Room Pacific Salon 1 - Session H3-1-WeM

Variable Temperature Nanomechanics
Moderators: Jeffrey M. Wheeler, ETH Zürich, **James Gibson**, RWTH Aachen University

8:00am **H3-1-WeM-1 On the Activation of Slip in the Mg-Al-Ca Laves Systems: A Combined Study Using High Temperature Indentation, Micropillar Compression and TEM***, **James Gibson**, C Zehnder, S Sandlöbes, S Korte-Kerzel,* RWTH Aachen University, Germany

The mechanical properties of modern, creep-resistant Mg-Al-Ca alloys are significantly influenced by the properties of the intermetallic skeleton at their grain boundaries. During application, these alloys will be thermally cycled from room- to application-temperatures, therefore it is essential to understand how the properties of these intermetallic components vary over this range.

A combination of nanoindentation, AFM, micropillar compression and TEM have been employed to study the behaviour of the Mg2Ca Laves phase between room temperature and 250˚C. Statistical analysis of the slip lines around intents - confirmed by TEM cross-sections - allow rapid analysis of relative CRSS values, supported by direct measurement in micropillar compression.

We show a constant hardness of ~3.5 GPa from room-temperature to 250˚C, revealing that Mg2Ca is likely the high-temperature strengthening phase of the parent alloy. The trends in slip-plane activation frequency and CRSS with temperature are analysed to explain the overall measurements of hardness.

8:20am **H3-1-WeM-2 Recent Evolution of Instrumentation for Nanoindentation Measurements at Elevated Temperatures***, **Philippe Kempe**, V Haiblíková,* Anton Paar, Switzerland

Characterization of thin film mechanical properties at elevated temperatures has been of scientific and industrial interests for many years, and Instrumented Indentation Testing (IIT) on PVD coatings is bringing useful information. The major limitations in high temperature measurements have been seen as the thermal drift, signal stability (noise) of instrumentation and oxidation of the surface. A defined setup of instrumentation allows to reduce these factors.

The vacuum nanoindentation system is designed to perform reliable load-displacement measurements over a wide temperature range (up to 800 °C). Vacuum has become an essential part of the instrument in order to prevent sample/tip oxidation at elevated temperatures. Independent tip and sample heating as well as an active thermal management of the system answer to the concern of temperature stability. Nevertheless, different experimental aspects of instrumentation are still investigated. It includes frame compliance, indenter tip calibration and verification, and reference samples. The manufacturing of indenter tips and their stability with temperatures is also discussed.

Recent measurements at high temperatures with system characterization and experimental protocol will be presented.

8:40am **H3-1-WeM-3 High Temperature Mechanical Characterization of Binary Cu-X Alloys Produced by Combinatorial Synthesis***, **Viswanadh Gowtham Arigela**,* Max-Planck Institut für Eisenforschung, Germany*; T Oellers, A Ludwig,* Ruhr Universitat Bochum, Germany*; C Kirchlechner, G Dehm,* Max-Planck Institut für Eisenforschung, Germany

Due of their excellent electrical properties copper-based material systems form the metallization components of most of the thin-film circuits today. The current trend of ever harsher environments and power densities brings the need of enhanced electrical and mechanical properties. It is of particular interest to develop copper alloys with improved strength, which requires the mechanical characterization of these systems at their service conditions on a micrometer length scale. We have used combinatorial material synthesis approaches to synthesize binary Cu-Ag and Cu-Zr alloys with the aim of enhancing the mechanical properties while preserving the electrical properties. The mechanical properties of the alloys were investigated by fabricating free-standing tensile specimens with photolithography techniques from the thin-film material libraries, which were produced by sputtering. Our approach enables high throughput mechanical characterization of a composition range of Cu-(1-8%) X. The alloys were tested both in the as deposited and in the annealed state. In addition, mechanical properties were also investigated at elevated temperatures (400°C) by tensile testing with a micro deformation stage with a novel method of temperature measurement. The investigations show a substantial improvement of the thin film strength both at elevated and at room temperature along the compositional gradient and a mild influence on the thin film conductivity. Beside the testing protocol and results we will also discuss the mechanism based origin of this behavior with respect to the thin film microstructure.

9:00am **H3-1-WeM-4 Temperature and Strain-rate Dependence of the Mechanical Behavior of Freestanding Gold Thin Films***, **Benoit Merle**,* Friedrich Alexander-University Erlangen-Nürnberg (FAU), Germany

The plastic deformation of freestanding gold films is shown to strongly depend on the testing temperature and strain-rate. These findings were achieved by both creep and constant strain-rate tensile tests, which were performed on evaporated gold films with columnar microstructure. The creep tests were carried out with an upgraded bulge tester operated between 23°C and 100°C. The measurements evidenced a critical temperature of about 75°C, corresponding to a transition in deformation mechanisms from a dislocation based to grain boundary and diffusion mediated plasticity. The influence of the stress was found to be rather low within the investigated range. Constant strain-rate tests were performed in-situ in a TEM, using a novel method for preparing tensile specimens from evaporated thin films. With decreasing strain-rate, the films exhibited a clear transition from shear-coupled grain boundary migration and grain growth to grain boundary sliding, which resulted in strong changes in strength and ductility.

9:20am **H3-1-WeM-5 In-situ Investigation on Mechanical Properties at the Micrometer Scale in Cryogenic Environment***, **Seok-Woo Lee**,* University of Connecticut, USA **INVITED**

Due to the significant advances in nanotechnology, a structural material at small length scales is becoming more important to develop mechanically robust small devices such as micro-/nano-electro-mechanical systems (MEMS/NEMS). MEMS and NEMS sensor systems that operate in the presence of high/low temperature, corrosive media and/or high radiation can reduce weight, improve machine reliability, and reduce cost in strategic market sectors such as automotive, avionics, oil well logging, nuclear power, and space exploration. Performance of all these small mechanical devices is directly related to mechanical properties of structural materials at small length scales, which are usually “different” from mechanical properties at bulk scale. In order to design a mechanically reliable small device working under various environments, therefore, it is critical to understand “how sample dimension influence mechanical properties of materials” as well as “how environmental conditions influence small-scale mechanical properties.” For the last two decades, small-scale plasticity has been extensively investigated by using micro-mechanical tests, and “Smaller is Stronger” and “Smaller is More Ductile” phenomena were observed in various material systems. As briefly mentioned before, the development of sensors and actuators that operates in harsh environment brings a strong attention in small-scale plasticity community. Therefore, materials research, which combines both “size effects” and “environmental effects”, is now regarded as next generation research in the field of materials science.

In this presentation, we are going to introduce our ongoing efforts to develop an in-situ nanomechanical testing system operating in cryogenic environments and describe its potential use for materials science research. Then, we will present several examples showing how temperature influences mechanical behaviors of materials at the micrometer scale. The size effects in body-centered-cubic single crystalline metals in cryogenic environments will be discussed, and the strong temperature dependence of power-law exponent will be explained. The size effects in metallic glassy nanolattices will also be presented. Here, we will discuss how thickness of metallic glass and temperature controls ductile-to-brittle transition in nanolattice structures. Finally, we will introduce our recent discovery of superelasticity in $ThCr_2Si_2$-structured intermetallic compounds, and their strong temperature dependence on their superelastic performance and structural transition will be discussed. Their potential use in cryogenic actuators or superconductivity switches for space exploration will be explained, too.

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