Advanced Characterization Techniques for Coatings, Thin Films, and Small Volumes
Room Pacific D - Session H2-1-TuM

Advanced Mechanical Testing of Surfaces, Thin Films, Coatings and Small Volumes I

Moderators: James Gibson, RWTH Aachen University, Germany, **Olivier Pierron**, Georgia Institute of Technology, USA

8:00am **H2-1-TuM-1 Reflectance Anisotropy Spectroscopy and Microscopy for the Investigation of Ultrathin Films With Micron Resolution***, Ralph Spolenak (spolenak@mat.ethz.ch),* ETH Zurich, Switzerland **INVITED**

Raman microscopy is the lab-scale gold standard for resolving stress distributions at the micron length scale. Unfortunately, the Raman effect is limited to polarizable materials, which typically excludes metals, an important materials class for both optical and plasmonic applications. Reflectance anisotropy spectroscopy (RAS) is, as our group recently demonstrated, sensitive to the effect of elastic distortion on the band structure of metals and thus allows for the determination of elastic strain.

While this is already valuable as an averaging technique further insight in both the plastic and brittle behavior of metals can only be gained on the local scale. Consequently, this contribution focuses on the development of a broad band RAS microscope, with the challenges of the broadening of the incidence angle as well as the accessible wave length range.

Our group is proud to present first results on pattering induced strain fields in gold thin films, the analysis of strain-engineered semiconductor devices and the observation of single plasmonic dipoles.

8:40am **H2-1-TuM-3 Combinatorial Mechanical Microscopy Using Correlated Nanoindentation Mapping and EDX***, Jeffrey M. Wheeler (jeff.wheeler@femtotools.com),* FemtoTools AG, Switzerland

Mechanical microscopy is an emerging technique using high-speed nanoindentation to map the mechanical behavior and extract phase-level properties from complex microstructures with micron-scale lateral resolution. As such, it is a powerful technique for combinatorial materials science investigations on samples with compositional gradients, such as diffusion couples. In this work, correlated high-speed nanoindentation and energy-dispersive spectroscopy (EDX) were applied to investigate the Ni—Ta system. All seven phases in the system were clearly resolved in the resulting maps, and the mechanical properties and composition ranges for each phase were determined. Good agreement with *ab initio* calculations was generally observed with some exceptions, most notably $NiTa_2$. This was achieved using a simple correlation method utilizing directly overlaid data matrices to allow compositional labeling of mechanical data. This allowed easy data segmentation without requiring complicated statistical deconvolution methods. Without this correlative method, phase deconvolution of the Ni—Ta system would have been challenging due to several phases possessing adjacent compositions and mechanical properties. This demonstrates the potential of this new correlative approach for future investigations, particularly those involving complex microstructures and/or compositional variation.

9:00am **H2-1-TuM-4 Progress in the Development of High Strain Rate Nanoindentation Experiments***, Warren Oliver (warren.oliver@kla.com),* KLA Corporation, USA*; C. Walker, B. Hackett,* Texas A&M University, Department of Materials Science & Engineering, USA*; P. Sudharshan,* International Advanced Research Centre for Powder Metallurgy & New Materials (ARCI) , India*; G. Pharr,* Texas A&M University, USA

High strain rate mechanical testing using instrumented indentation has recently received considerable attention. High strain rates as high as 10^4 are achievable at reasonable indenter velocities (on the order of 0.5 m/s) because the sample size is small. An instrument capable of displacement measurements at rates as high as 1.25 Mhz with sub nanometer resolution and a time constant of 10 microseconds has been constructed. The system also incorporates mechanics that allow the load frame to have a stiffness as high as 90 MN/m making the associated correction very small. While the system directly measures the displacement signal needed accurately, measuring the loads exerted on the sample in sub millisecond experiments can be challenging. In some cases, the time constant of the loading system must be considered. In addition, the load can be dominated by inertial effects. Dynamic models used to describe the system will be considered. A number of approaches for calculating or measuring the loads exerted on the sample will be discussed. Preliminary data for fused silica and Aluminum will be presented.

9:20am **H2-1-TuM-5 Testing the Adhesion of a Sintered Ag Film on a Cu Substrate Using Laser Shocks***, Xavier Milhet (xavier.milhet@ensma.fr),* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France*; T. de Resseguier,* institut pprime - CNRS - ENSMA - Université de Poitiers, France*; A. Sghuri,* Institut Pprime - CNRS - ENSMA - Université de Poitiers, France*; L. Signor,* institut pprime - CNRS - ENSMA - Université de Poitiers, France

Silver (Ag) paste sintering, is now used for die bonding in the latest generation of power electronic modules. Since the processing conditions require low temperature, low stress and short time, the Ag joints exhibit a porous structure that evolves significantly when exposed to the operating temperatures (between 250C and 300C) [1,2]. In order to model the behavior and properties evolution, there is a need for a fine characterization of the joint itself as well as those of the interface, especially during aging. While the first point has now been relatively widely investigated [3-5], the information on the interface properties is still scarce. In this study, we have explored an alternative route to test the adhesion of the Ag film on a copper (Cu) substrate using laser driven shocks: a laser pulse is calibrated to induce tensile loading near the interface between the substrate and the film, and time-resolved velocity measurements complemented by post-recovery observations provide quantitative information on the adhesive strength. Although such highly dynamic load is somewhat far from the practical application, this method presents the advantage over other techniques to really focus on the interface properties. The results are used to explore the relationship between adhesion, aging and the underlying microstructure.

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4 - X. MILHET, P. GADAUD, V. CACURRI, D. BERTHEAU, D. MELLIER, M. GERLAND Journal of Electronic Materials 44 (2015) 3948

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9:40am **H2-1-TuM-6 Transfer Learning in Characterization of Nanoindentation Induced Acoustic Events***, Antanas Daugela (antanas_daugela@hotmail.com),* Nanometronix LLC, USA*; J. Daugela,* Johns Hopkins University, USA

A passive monitoring of acoustic waves during nanoindentation has been attracting the attention of material scientists since the inception of nanomechanical test instruments. The conventional acoustic wave signal treatment via RMS or integrated energy values proved that quantitative acoustic wave properties correlate well with the local contact materials' phenomena such as yield point initiation for W(100) [1, 2], Sapphire [3], phase transformations on SMA, and differentiating of thin film fracture modes.A nanofatigue phenomenon can be observed on thin films by monitoring the resulting multi-cycle nanoindentation loading-unloading curves and post test imaging, which helps in identifying the materials' phenomena [4].However, the true potential of the acoustic characterization method is unleashed in the synergy between wavelet based acoustic signal decomposition and machine learning [5].

The Transfer Learning is a subclass of machine learning which utilizes existing Deep Learning Neural Networks [5].In this work, a Transfer Learning based signal classification of nanoindentation induced passive and active acoustic events is explored.Both passive and active acoustic monitoring can be conducted during nanoindentation with the integrated utrasonic tip.The proposed Transfer Learning technique yields a reliable classification of acoustic signatures on submicron thick coatings.

References:

1. A. Daugela et al, *Zeitschrift fur Metallkunde*, **92(9)**, p.1052-1056 (2001)

2. N. I. Tymiak et al, *Journal of Materials Research*, **18(4)** , p.1–13 (2003)

3. N.I. Tymiak et al, *Acta Materiallia*, **52** p.553-563 (2004)

4H. Kutomi et al, *Tribology International*, **36**, p.255-259 (2003)

5. A. Daugela et al, *Materials Science & Engineering A*, **800** 140273 (2021)

10:00am **H2-1-TuM-7 Nanoindentation Testing to Measure Surface Free Energy in Thin Films and Engineered Surfaces**, *M. Sebastiani,* Università degli studi Roma Tre, Italy*; P. Phani,* International Advanced Research Centre for Powder Metallurgy & New Materials (ARCI), India*; **Edoardo M. Rossi (edoardo.rossi@uniroma3.it)**,* Università degli studi Roma Tre, Italy*; R. Guillemet,* Thales Research & Technology, France*; W. Oliver,* Nanomechanics Inc., KLA Corporation, USA

The ability to engineer and control the Surface Free Energy (SFE) of functional materials is critical for a multitude of applications as this property represents a relevant design parameter for producing components with precisely controlled interfacial performances. The non-destructive measurement of SFE in nanopatterned superhydrophobic hard surfaces is a challenge in both research and industry since, in most cases, time-consuming contact angle measurements (CAMs) are not feasible. Nanoindentation testing can offer a possible contact-mechanics methodology that allows measurement of intrinsic surface properties [1]. In this sense, a novel method has been developed for the assessment of the adhesive interactions by carefully controlling environmental and instrumentation issues. A commercially available nanoindenter has been specifically modified to enable accurate measurement of the pull-off forces raising between the tip and the sample surface. A novel testing protocol implementing adhesion-accounting contact mechanics models [2] has been developed to leverage the new hardware capabilities.

A set of reference surfaces was selected for testing and validation: (i) highly energetic, new, and atomically flat surfaces from cleavage of the silicate sheet-based structure of muscovite mica; (ii) DSP germanium <100> crystals. The latter yielded the pristine substrates for the development of superhydrophobic surfaces, via both patterning and fluorinate silane coating. Those processes performances were independently studied and, ultimately, their interplayed role was investigated on both nanopatterned and silanized substrates. CAMs were performed on all the surfaces investigated to provide comparative results. The method is found to measure SFE over five orders of magnitude, covering hydrophilic to super-hydrophobic surfaces.

[1] F. M. Borodich et al. Proc. R. Soc. A. 464, 2759–2776 (2008).

[2] K. L. Johnson et al. J. Colloid Interface Sci. 192, 326–333 (1997).

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