Thin Films
Room 306 - Session TF-WeM

Mechanical and Tribological Properties of Thin Films
Moderator: B.C. Holloway, College of William and Mary

8:20am **TF-WeM1 Mechanical Properties of Thin Films and Multilayers**, ***F. Spaepen,*** Harvard University **INVITED**

Thin films and multilayers are ideal systems to study the dependence of the mechanical properties on various length scales: grain size, film thickness, layer thickness, compositional modulation and dislocation cell size. Special techniques are required to perform tensile tests on thin films, which can be free-standing, or in the case of metallic films less than 1 mm thick, supported on thin, compliant polymeric substrates, such as Kapton. These techniqes allow determination of stiffness, anelastic behavior, yield stress, work hardening and fracture, all of which depend in characteristic ways on the various length scales. The development of stress in a thin film during deposition can be studied by measuring the curvature of the substrate (usually by laser reflection, often in situ) or the average lattice parameter (by X-ray diffraction). The development of the stress is strongly affected by the evolution of the microsctructure. Interfaces play a particularly important role. For example, the trade-off between surface and grain-boundary energies provides the (tensile) strain energy in island coalescence. Examples will be given from metallic (Cu, Ag,..) and ceramic (YSZ) films.

9:00am **TF-WeM3 Evaluation of Applied Substrate Bias on Yttria-Stabilized Zirconia Thin Films**, ***J.R. Piascik,*** University of North Carolina at Chapel Hill and RTI International; *J.Y. Thompson,* University of North Carolina at Chapel Hill; *C.A. Bower,* RTI International; *B.R. Stoner,* RTI International and University of North Carolina at Chapel Hill

Partially stabilized zirconia (PSZ), possesses a unique set of material properties. Due to its high toughness, excellent wear behavior, and thermal stability, PSZ, in the form of thin films, can be used for a variety of applications ranging from biomedical to thermal barriers. The objective of this research was to study environmental effects on film stress and model water absorption into the defect structure of films deposited with varying substrate bias. Yttria (3 mol%)-stabilized zirconia (YSZ) thin films were deposited using radio frequency (RF) magnetron sputtering. YSZ thin films were deposited at a working pressure of 15mT, temperature of 150°C, and Ar/O2 gas ratio of 30:1. An applied substrate bias was varied (0 to 50W) to alter film density and structure. X-ray diffraction (XRD) showed that films increased in monoclinic phase percentage as a function of increasing substrate bias power. Wafer bow measurements indicate that initial film stress increased in a compressive direction (70 to 302MPa) as substrate bias was increased. Aging in ambient environments (25°C, 75% relative humidity) led to a significant increase in compressive stress (80MPa) for films deposited without substrate bias. Thermal treatment, to remove absorbed water, caused films to return to near-initial stress states. Once exposed to ambient conditions, measured stress was found to increase at a rate of 0.20MPa/min for the first 2hr of exposure then by approximately 2MPa/day for a period of 30 days. Films deposited with applied substrate bias displayed a reduction in this time-dependent phenomenon. Cross-sectional TEM allowed for high-resolution images to analyze film structure and defect density. A subsequent model is proposed, describing the incorporation of water vapor into structural defects in the deposited films. This work is supported through NIH-NIDCR R01 DE013511.

9:20am **TF-WeM4 Ion Beam Assisted Deposition of Cubic Zirconia**, *X. Wang,* University of Nebraska-Lincoln; *I. Amirani, S. Varma,* University of Nebraska Medical Center; *D.W. Thompson,* University of Nebraska-Lincoln; *F. Namavar,* Universoty of Nebraska Medical Center; ***N.J. Ianno,*** University of Nebraska-Lincoln

Cubic zirconia's excellent wear properties and inertness make it an excellent choice for a range of applications. In this work we will show that ion beam assisted deposition (IBAD) of thin film single phase cubic zirconium oxide has been accomplished. Deposition is accomplished by electron beam evaporation from a pure zirconium oxide source in a low pressure oxygen ambient coupled with simultaneous argon ion beam bombardment of the growing film. We will show that by proper control of the deposition parameters the grain size can be varied in a continuous manner from micron scale at the film-substrate interface to the nanometer scale at the film surface. This microstructure results in a film with an extremely high hardness. The deposited films will be characterized by x-ray diffractometry, atomic force microscopy, interferometry, transmission electron microscopy and spectroscopic ellipsometry.

9:40am **TF-WeM5 Effects of TiO@sub x@ PVD Deposition Parameters on the Preferred Orientation and Adhesion of Pt Films on @gamma@-Al@sub 2@O@sub 3@**, ***E. Derniaux,*** ONERA France; *P. Kayser,* ONERA France, FRANCE; *C. Gageant, C. Sanchez, D. Boivin,* ONERA France

PVD TiO@sub x@ films are studied in order to improve the adhesion of Pt/AlN/Pt piezoelectric transducers on @gamma@-Al@sub 2@O@sub 3@/NiCoCrAlY/superalloy components. Thick (2 µm) TiO@sub x@ films deposited by RF reactive cathodic sputtering on @alpha@-Al@sub 2@O@sub 3@ substrates were analysed by X-ray diffraction (XRD) and scanning electron microscopy equipped with X-ray spectroscopy (SEM/EDX). The influence of substrate temperature, O@sub 2@/(Ar+O@sub 2@) ratio in the plasma gas and annealing (1050°C, 1h, in air) on structural properties, morphology and chemical composition was observed. Thin (@<=@100 Å) TiO@sub x@ adhesion layers and Pt films were sputtered on @alpha@-Al@sub 2@O@sub 3@. The effects of TiO@sub x@ deposition parameters on the adhesion and the orientation of as-deposited and annealed Pt films were examined respectively by pull off test and XRD. Results showed the (111) preferred orientation and the good adhesion of Pt films. Finally, the selected TiO@sub x@ deposition parameters were applied on @gamma@-Al@sub 2@O@sub 3@/NiCoCrAlY/superalloy substrates. Adhesion properties of annealed Pt films on @gamma@-Al@sub 2@O@sub 3@ and TiO@sub x@/@gamma@-Al@sub 2@O@sub 3@ were evaluated, the results showed a drastic improvement of the pull off force (0 to 1000 kg.cm@super -2@) by using the TiO@sub x@ 'glue layer'. The adhesion layer led also to a higher (111) preferential orientation of the Pt films.

10:00am **TF-WeM6 Microstructure and Stress of Room Temperature Reactively Sputtered RuOx Thin Films**, *J. Shi,* ***T.M. Klein,*** The University of Alabama

RuOx thin films were deposited at room temperature by reactive RF magnetron-sputtering using Ar/O2 discharges of varying O2 flow ratio (O2/Ar+O2) over the range 10% to 50%. With an increase of O2, the film texture changed from (110) to (101). Films deposited with a flow ratio >25% were determined stoichiometric. Apparent grain sizes, densities and hardnesses by nanoindentation were measured as a function of flow ratio. Deposition rate, resistivity and intrinsic stress trends with O2 flow ratio were similar. The intrinsic stresses in as-deposited films were all compressive and increased with addition of O2, except for the film sputtered at a flow ratio of 20% which was in biaxial tension. Stress-temperature behavior during a thermal cycle in air up to 500ÂºC changed for films deposited at different flow ratios due to varying microstructure evolution. The film deposited at a flow ratio of 30% became nearly stress-free with a low resistivity value of 68 micro Ohm-cm after anneal which is promising for use in micro-devices.

10:20am **TF-WeM7 Synthesis of Carbon Films with Ultralow Friction in Dry and Humid Air**, ***C.A. Freyman,*** *Y.F. Chen, B. Zhao, Y.W. Chung,* Northwestern University

In this paper, we present experimental strategies of how one can synthesize smooth carbon films using sulfur doping with ultralow friction coefficients (µ< 0.01) in dry and humid air, with relative humidity up to 50%. Auger studies show the absence of water adsorption after room temperature water exposure of 100,000 Langmuirs. Thermal desorption studies suggest the formation of water multilayers even at very low exposures, indicating that the surface is hydrophobic, consisting with water contact angle measurements. Results from quartz crystal microbalance studies at ambient pressures will also be discussed.

10:40am **TF-WeM8 Surface Modification of Si@sub 3@N@sub 4@ Probes for Adhesion and Wear Reduction**, ***Z. Tao,*** *B. Bhushan,* The Ohio State University

Tip wear of Si@sub 3@N@sub 4@ probes used for atomic force microscope (AFM) can result in increase of tip radius, thus reducing the image resolution and introducing artifacts. Adhesion between the tip surface and sample is another source of image artifacts. In order to reduce adhesion and wear, perfluoropolyether (Z-TETRAOL) and Fluorinert@super TM@ will be deposited on the Si@sub 3@N@sub 4@ probe. The adhesion, friction, and wear of the uncoated/coated tips will be investigated. The humidity effects on the adhesion, friction, and wear of the uncoated/coated tips will be investigated as well. The influence of the coating on the image resolution will be discussed.

11:00am **TF-WeM9 Ultra-Hard Al-Si Nanocomposites Synthesized by High-rate Co-evaporation**, ***D. Mitlin**, C. Ophus,* University of Alberta, Canada*; V. Radmilovic, T.J. Richardson, U. Dahmen,* Lawrence Berkeley National Laboratory

We used high rate co-evaporation to synthesize Al-Si nanocomposite films in the composition range of Al-1at%Si to Al-23at.%Si. Pure Al films were also synthesized using identical deposition conditions, and were used as a baseline. The Al-Si structures have a hardness as high as 4GPa (Al-23at.%Si), and display noticeable plasticity under nanoindentation (Al-12at.%Si). We used transmission electron microscopy (TEM) to analyze Al-12at.%Si and pure Al samples. The pure Al film had the expected grain size of upwards of one micrometer. The Al-Si film consists of a dense distribution of spherical nanoscale (10-30 nm) Si particles separating irregularly-shaped Al grains, many of which are also nanoscale (50-200 nm). This microstructural refinement is due to the Si particles disrupting the columnar growth of the Al grains by promoting re-nucleation. Additionally the particles also pin the Al grains which coarsen during deposition. X-ray diffraction results indicate that there is a marked decrease in the Al lattice parameter with increasing Si content. This effect is weaker after the samples have been stored at room temperature for six months. The structures' electrical resistivity remains near the level of pure Al for compositions up to 6.5at.%Si, but significantly increases at higher Si compositions. This work is the first step towards yielding Al-Si nanomaterials specifically optimized for integration into structural components of MEMS.

11:20am **TF-WeM10 Hard Coatings of Tungsten Nitride Grown by Reactive Sputtering and Laser Ablation**, ***E.C. Samano**, A. Clemente, M.J. Oviedo, G. Soto,* CCMC-UNAM, Mexico

The search for materials showing chemical inertness and high hardness, elastic modulus and melting point is still intense. Transition metal nitride coatings are an alternative because they have been traditionally used as protective coatings against wear and corrosion. Due to the fact that refractory metals are hard materials with a high melting point, tungsten nitride coatings are an excellent choice. Their properties can be tailored by tuning the nitrogen content during film synthesis. The investigation on the relationship between thin film preparation conditions and mechanical properties for tungsten nitride films is not as well understood as other transition metal nitrides, like titanium nitride. We report the growth of tungsten nitride films grown by two different deposition methods, reactive sputtering and laser ablation, in the ambient of N@sub 2@ at various pressures on stainless steel substrates. The composition of the films is determined by AES and XPS. The stoichiometry of the coatings grown by reactive sputtering are found to be W@sub2-x@N, 0@<=@x@<=@0.5, while those grown by laser ablation are W@sub2@N, WN and WN@sub2@. The mechanical properties of the films grown by both deposition methods are studied as a function of N@sub2@ pressure. The hardness of the deposited films resulted to be in the 25 to 35 GPa range.

11:40am **TF-WeM11 Electron Spectroscopic Studies of Friction Modifier Thin Films**, ***K.C. Wong**,* University of British Columbia, Canada*; X. Lu,* Kelsan Technologies Corp.*; P.C. Wong,* University of British Columbia, Canada*; J. Cotter, D. Eadie,* Kelsan Technologies Corp.*; K.A.R. Mitchell,* University of British Columbia, Canada

Friction modifier (FM) thin films are important for controlling friction at the wheel-rail interface of trains. Products on the market have progressed to date largely by empirical development. Accordingly we have initiated a program to investigate how FM model systems are affected by tribological experiments that simulate wheel-rail contact using a rolling disk-on-disk setup adjusted for designated pressure and creepage. This paper will report spectroscopic characterizations (with XPS, Raman and SAM) and thermogravimetric analyses for FM materials (e.g. MoS2, graphite, BN, WS2, ZnO, MoO3, talc, SnO2, polytetrafluoroethylene, polyimide and polyamide) and water-based emulsion polymers as binders, where the studies are made as a function of coefficient-of-friction, number of rolling cycles and temperature.

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