Thin Films Division
Room 310 - Session TF-MoA

Mechanical Properties of Thin Films

Moderator: F.K. Urban, Florida International University

2:00pm **TF-MoA1 Computer Simulation Modeling of Sculptured Thin Films**, ***V.C. Venugopal***, *R. Messier,* Pennsylvania State University

Sculptured Thin Films (STFs) are characterized by free standing columns whose shape can be engineered as desired. Films with S-shaped, C-shaped, helicoidal or chevronic columns have been grown. Low adatom mobility and self-shadowing effects are critical for the growth of such films. To aid characterization of STFs theoretically, the growth of STFs is simulated using a simple ballistic aggregation deposition model assuming a high sticking coefficient, negligible relaxations, and low substrate temperatures. Substrate manipulations and complex substrate topographies are investigated. Clustering is found at the 1-3 nm level and is related to the larger column sizes which result from competitive growth evolution. A 3-D model of a growing STF is built up. The model structures are directly correlated to our experimental results in the relations between the incoming vapor angle @chi@@sub v@, and the resulting morphology column angle @chi@@sub m@. The final simulated structure is being used to develop mechanical models of STFs. Mechanical strength and elastic moduli can be determined at several different continuum levels and verified experimentally to develop a reliable model. Residual stress fields and other mechanical characteristics can also be studied. Using acoustic wave propagation principles, the maximum theoretical strength of these films can be determined and verified experimentally. Initial results of such models developed are presented.

2:20pm **TF-MoA2 Characterization of TiN/CN@sub x@ Multilayers Deposited by DC Magnetron Sputtering**, ***M.M. Lacerda***, *Y.H. Chen,* Northwestern University; *W.C. Chan,* University of Hong Kong, China; *B. Zhou, Y.W. Chung,* Northwestern University

Titanium nitride (TiN) is commonly used in wear protection coatings due to its high hardness. However, it is well known that thick TiN films develop columnar structure and are subject to cohesive failure. In this work, we used CN@sub x@ thin films to interrupt the growing TiN before the columns could initiate. The samples have been deposited by DC unbalanced magnetron sputtering at low pressure (2.5 mTorr) using an argon-20% nitrogen mixture. We applied a substrate bias of -200 V to promote ion bombardment. The CN@sub x@ thickness was kept constant at 1.3 nm. X-ray diffraction (XRD) patterns obtained at low angles (2°@<=@2@theta@@<=@5°) showed good interface between layers. XRD patterns at higher angles showed strong TiN (111) texture. The mechanical properties of the multilayers have been studied as a function of the TiN/CN@sub x@ thickness ratio (t@sub r@). Nanoindentation of samples with t@sub r@ = 2.3 showed high hardness value as compared to TiN films deposited at the same conditions. The TiN/CN@sub x@ films are at least 2.5 times harder than TiN samples. Internal stress of the same samples was calculated by the substrate curvature. Results showed that multilayers have compressive stress up to 10 times lower than TiN films. Electron microscopy results of the microstructure of these multilayered coatings will be presented.

2:40pm **TF-MoA3 Characterization of Stress-Morphology Relationships in Sculptured Thin Films (STFs)**, ***R.A. Knepper***, *D.E. Fahnline, R. Messier,* Pennsylvania State University

Sculptured Thin Films (STFs) are a recent development in thin film technology wherein a substrate is rotated while a columnar thin film is deposited at varying oblique angles and orientations. The resulting microstructure can thus be engineered into a number of shapes, including non-normal matchsticks, zigzags, coils, and periodically bent nematics (S- and C-shapes). STFs are highly porous and can have properties that differ greatly from both the bulk material and isotropic films of the same material. However, the nature of the relationships between STF morphology and intrinsic deposition stress is not yet understood. In this work, a set of matchstick-shaped STFs has been prepared with varying vapor incidence angles, ranging from 15° 75° from the substrate surface. The deposition results in curvature changes along the two directions of the substrate surface that have been measured by a laser scanning method. These measurements are then used to calculate the biaxial stresses in the films. The measured stresses decrease with decreasing morphology angle, with an abrupt change at a vapor incidence of 45°, and are anisotropic with respect to direction. The stresses measured ranged from 60 MPa to 0.4 MPa. The implications of these results to the general sets of STFs will be discussed. The findings of this research may be used to evaluate future models of the origins of stress in STFs, as well as to control substrate curvature and avoid delaminations.

3:00pm **TF-MoA4 Mechanical Properties Measurements using Scanning Force Microscopy**, ***W.N. Unertl***, University of Maine **INVITED**

Considerable effort is aimed at using the Scanning Force Microscope (SFM) to measure the mechanical properties of surfaces with nanometer-scale resolution. The properties of interest include the Young and shear moduli, shear strength, and work of adhesion. The most widely used approach is to extract these properties from the SFM data by simply scaling the results of macroscopic continuum contact mechanics theory to the dimensions and forces of an SFM contact. This talk will focus on two aspects of this scaling problem@footnote 1@: (1) the mechanism for failure of a contact under shear and (2) the effect of creep on contacts to viscoelastic materials. In a contact subjected to a shear strain, contact mechanics predicts that a crack propagates at the interface and causes a non-linear increase in shear force until the interface ruptures and sliding begins. This behavior, called microslip, is observed for macroscopic contacts but not for SFM contacts, which suggests that the contact mechanics picture must be modified for nanometer-scale contacts. In contacts to viscoelastic materials, creep can significantly modify the formation and rupture of a contact compared to contacts to elastic materials. The most important effect is that the maximum contact area depends on the loading history and, unlike elastic materials, can reach its maximum value well after the maximum load is applied. The status of theoretical models for the analysis of contacts to linear viscoelastic solids including the effects of adhesion will be described. @FootnoteText@ @footnote 1@K.J. Wahl, S.V Stepnowski, W.N. Unertl, Tribology Lett. (in press 1998).

3:40pm **TF-MoA6 Meso-Scale Contact Hardness, Friction, and Wear of Aluminum Oxynitride Films**, ***S.D. Dvorak***, *G.P. Bernhardt, O.D. Greenwood, R.J. Lad,* University of Maine

Aluminum oxynitride (AlO@sub x@N@sub y@) thin films attract interest as hard, wear resistant coatings for high temperature, oxidizing environments. We have used electron-cyclotron-resonance (ECR) plasma assisted electron beam evaporation of aluminum to grow aligned crystalline films on r-sapphire at 800 - 1100 K to nominal thicknesses of 100 nm at about 0.5 Å/s deposition rates. These AlO@sub x@N@sub y@ films were fabricated with compositions ranging from aluminum oxide to aluminum nitride, depending on the N@sub 2@/O@sub 2@ gas flow ratio in the ECR source. Film hardness as a function of depth was measured by nanoindentation, while friction and wear properties were determined during reciprocal sliding experiments using well characterized sapphire and diamond probe tips with applied loads in the micro-newton to milli-newton force range. Film topography examined with atomic force microscopy indicated rms roughness values ranging from 20 Å to 140 nm. Wear tracks examined by AFM consist of wear debris as well as microstructural features. We observe that friction is affected by the roughness of the surfaces in contact, and that these roughness effects are dependent on the hardness of the contacting asperities, as measured by a Hysitron Pico-Indentor. Inhanced surface diffusivity of oxygen and nitrogen species provided by the ECR source during film growth yields highly-oriented films with very high wear resistance.

4:00pm **TF-MoA7 Nanotribology of Single Crystal ZnO Surfaces: Relation of Atomic Level Friction to Macro Tribology of Thin Films**, ***J.J. Nainaparampil***, *J.S. Zabinski, S.V. Prasad,* Air Force Research Laboratory

Atomic Force Microscopy (AFM) has been applied to the study of surface forces for more than a decade. Relatively recently, Lateral Force Microscopy (LFM) has evolved from AFM as a means to characterize surface forces in relation to friction, adhesion and surface topography. The significance of this approach is that it reveals insights into friction and wear at an atomic level. This work focuses on the nanotribology of single crystal ZnO surfaces after high temperature annealing treatments and in different gases. Annealing causes the formation of surface structures - etch pits on the 1010 surface and roughening or reconstruction on the 0001 surface. The pits and roughened areas provided lateral force contrast that could not be assigned to topography. Adhesion and relative contact stiffness were not significantly different among friction contrasting regions. The chemistry of these regions was analyzed using Scanning Electron Microscopy (SEM) and Scanning Auger Microscopy (SAM). The LFM and chemical analysis of the different single crystal surfaces will be presented. Insights into atomic level friction and wear processes will be related to the macroscopic tribology of ZnO nanocrystalline thin films.

4:20pm **TF-MoA8 Surface Stress in Silicon Oxide Layer made by Plasma Oxidation with Applying Sample Bias***, A.N. Itakura,* National Research Institute for Metals, Japan*; T. Kurashina, T. Narushima,* University of Tsukuba, Japan*; **M. Kitajima**,* National Research Institute for Metals, Japan

We present the evolution of surface stress during plasma oxidation of Si(100) with applying bias voltage from -60V to +60V to the sample. The experiments were performed in a UHV condition. Oxide thickness was controlled from 0 to 3nm. The sample was a cantilever of Si(100) of dimensions 450µm x 50µm x 4µm. The bending of the lever due to stress was detected as a function of the oxidation time by a change in the reflection angle of laser beam from lever backside. Stress was calculated from the lever deflection using Stony's formula. The plasma was generated by RF discharge of oxygen gas at 13.56MHz. There has been observed three stages in the stress vs. time curve for the plasma oxidation of Si cantilever without applying sample bias. The first stage was rapidly building up of tensile stress and the second stage was the tensile stress decreasing slowly. In the last stage the stress changed to compressive. For the cases of oxidation with applying bias to the samples, stress curves showed different time dependence from that without bias. First, the stress curve showed a quick build-up of compressive stress, followed by a tensile stress formation, and the stress gradually changed to compressive one with further oxidation. The similar feature appeared in the curves with positive biases and negative biases. The stress values were not unique at same thickness but strongly depended on a bias voltage. We will discuss these stress changes in terms of the interface structure of silicon-oxide layer and silicon substrate.

4:40pm **TF-MoA9 Stress Alignment in SiO@sub 2@ Thin Films Deposited on Thin Chromium and Aluminum Film***, **K.E. Coulter**, V. Raksha,* Flex Products, Inc.

SiO@sub2@ as a low index material in optical applications is often complicated by the intrinsic stress that induces film cracking, substrate deformation and delamination. 400 - 600nm thick SiO@sub2@ films were deposited by e-beam evaporation onto Cr (10nm) and Al (100nm) films. Using design of experiment methodology, we evaluated deposition process parameters such as rate, vacuum pressure, substrate type, coating material and storage conditions. The thin films were deposited on 50 and 175 µm thick PET substrates as well as fused silica witnesses. Analysis methods included interferometry, profilometry, microscopy, ellipsometry and an evaluation principle based on laser deflection off the free end of a coated PET strip. A correlation was established between absolute stress values measured by interferometry and the laser deflection method. Cr/ SiO@sub2@ and Al/ SiO@sub2@ films were deposited under vacuum conditions which produced tensile and compressive stress. S! tress in all SiO@sub2@ thin film stacks became more tensile with age (shelf life) regardless of the initial stress in the film. Dopants in the silica source material such as B@sub 2@O@sub 3@, Na@sub 2@O and Vycor produced films with similar correlations between stress and deposition conditions but at lower absolute stress magnitudes. In this presentation, the influence of the vacuum pressure, deposition rate, storage conditions and substrate properties will be discussed relative to the effect of stress on the thin film optical performance.

5:00pm **TF-MoA10 Investigation of Induced Recrystallization and Stress in Close-Spaced Sublimation CdTe Thin Films***, **H.R. Moutinho**, R.G. Dhere, M.M. Al-Jassim, P. Sheldon,* National Renewable Energy Laboratory*; B.T. Mayo,* Southern University*; L.L. Kazmerski,* National Renewable Energy Laboratory

Close-spaced-sublimation (CSS) CdTe has produced the best CdS/CdTe thin-film solar cells reported to date. In all CdTe cell deposition options, a post-deposition treatment with CdCl@sub 2@/methanol solution at elevated temperature is a mandatory step for maximizing the device efficiency. We have previously reported that these large-grain CSS films do not recrystallize and that the initial in-plane stress is not completely relieved during the treatment, in contrast to films deposited by other methods (e.g. physical vapor deposition). In this work, we deposited CSS CdTe films at lower temperatures and higher deposition rates to force lower-grain-size layers, which are more susceptible to recrystallization. The objective was to induce recrystallization from the chemical/heat treatment to realize films with substantially less stress and, consequently, better device quality. The CdTe films were deposited on normal CdS/SnO@sub 2@/glass structures and chemically treated at various temperatures and times to optimize the recrystallization process. The topography and grain size of the films were determined by atomic-force microscopy, X-ray diffraction, and transmission electron microscopy, and the minority-carrier lifetime by time-resolved photoluminescence. The CdCl@sub 2@ treatment temperature was varied from 300 to 400° and the treatment time from 1 to 30 minutes. The stress in the films was investigated using X-ray techniques, and significant reduction in the stress was observed concurrent with the recrystallization/recovery process. We investigated the evolution of stress in the early stages of the treatment to establish the mechanisms through which recrystallization starts in these films. CdTe films deposited by physical vapor deposition were also analyzed, and the results were compared with the ones for the CSS films. Finally, the efficiencies of cells prepared from conventional CSS CdTe were compared to these lower stress thin-film devices.

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