Thin Films
Room 329 - Session TF-FrM

Mechanical Properties of Thin Films

Moderator: B.C. Holloway, College of William & Mary

8:20am **TF-FrM1 Island Coalescence Stress Created During Thin Film Growth**, ***S.J. Hearne***, *J.A. Floro, C. Dyck, T. Christenson,* Sandia National Laboratories; *W. Fan, S.R.J. Brueck,* University of New Mexico

For over 30 years it has been generally accepted that island coalescence during thin film growth results in a tensile stress. However, prior to this work there had been no conclusive demonstration of the fundamental physical mechanism underlying this process. Using selective lateral film growth on patterned substrates during electrodeposition of Ni films, we have obtained the first unambiguous comparison of the experimentally-measured tensile stress due to island coalescence with theoretical predictions. This technique allows for the systematic variation of island size and geometry while maintaining temporally constrains of the coalescence of Ni islands during electroplating. This avoided the complications associated with stochastic island coalescence that have plagued previous measurements obtained over the last 30 years. We found that the functional dependence of the stress on island size and dimensionality is consistent with a Hertzian contact model recently proposed by Freund and Chason.@footnote 1@ However, the absolute magnitude of the measured stress is smaller by factors of 2-4x, and reasons for the discrepancy will be discussed. This work was partially supported by the DOE Office of Basic Energy Sciences. Sandia is a multiprogram laboratory of the United States Department of Energy operated by Sandia Corporation, a Lockheed Martin Company, under contract DE-AC04-94AL85000. @FootnoteText@ @footnote 1@L.B. Freund, E. Chason, JAP 89, 4866 (2001).

8:40am **TF-FrM2 Alloying Effects on Mechanical Behavior of Thin Metal Films**, ***R.P. Vinci***, Lehigh University **INVITED**

Thin metal films such as Al, Pt and Au are often used in their pure state in microelectronics and MEMS applications. This is true despite the fact that their mechanical behavior is frequently critical for performance. When thin film alloys are employed, a specific alloy composition is often selected because it is a preexisting combination found in a particular fabrication facility, not because it is the optimum composition for the application at hand. In contrast, bulk metals are almost always alloyed for mechanical and microstructural stability, and the effects of various alloying elements are well characterized. Should a designer of microelectronics and MEMS applications need a particular set of mechanical properties (e.g., fatigue or creep resistance) there is no available equivalent understanding of alloy effects in thin film form. In this presentation, systematic experiments focused on elucidating the effects of alloying on thin film mechanical properties will be described. In particular, deviation from (or agreement with) bulk behavior will be discussed for several metal alloy systems.

9:20am **TF-FrM4 Mechanical Properties of Reacted Metal Multilayers**, ***D.P. Adams***, *M. Rodriguez, J.B. Kelley, T. Covert, T. Buchheit, M. Grubelich,* Sandia National Laboratories

Sputtered multilayer thin films composed of reactive material pairs are currently of interest for brazing, joining and other energetic applications. As shown extensively by Weihs et.al. several thin film material systems can be stimulated such that a rapid, self-propagating reaction occurs within a multilayer. This alloying reaction has great potential for joining, because it prevents global heating of parent materials. In this talk, we evaluate the resultant mechanical properties and microstructure of three different reacted multilayer systems. This includes Ni/Ti, Al/Pt and metal/B multilayers. Each material system was deposited by magnetron sputtering to thicknesses in excess of 1 ⍰-m. First, we describe how the propagation velocity depends on multilayer design. Propagation velocities are measured by imaging the propagating reaction front using a high speed Cordin camera and strobe light assembly. Velocities in excess of 50 m/s are measured for some multilayer designs. We determine how changes in stoichiometry (specifically deviations from the targeted composition) affect velocity. Secondly, we discuss the stress in reacted films. Laser-based wafer curvature techniques are used to determine average in-plane stress in reacted films. Stresses in excess of 1 GPa have been measured for several reacted multilayers. To better understand the development of stress in these systems we have conducted x-ray diffraction to determine coefficients of thermal expansion. Films achieve extremely high temperatures during self-propagating reactions and extrinsic stress largely determines final stress state; this can greatly affect their usefulness in a braze process. Additional mechanical properties such as hardness are measured, and these are related to film microstructure probed by transmission electron microscopy.

9:40am **TF-FrM5 Intrinsic Stresses in CrN Films Deposited by Arc Ion Plating**, *T. Hanabusa,* ***K. Kusaka***, Tokushima University, Japan; *T. Matsue,* Niihama National College of Technology, Japan

It is well known that large compressive residual stresses of the order of GPa develop in TiN and CrN films deposited by ion plating. The residual stress in the film is the sum of thermal stress and intrinsic stress. The former is the stress originated by the difference in the thermal contraction between the substrate material and the film, whereas the latter is originated by various factors during the depositing process. The residual stress in TiN or CrN film is predominantly the intrinsic stress. The object of this study is to investigate an essential meaning of the intrinsic stress in the films. In this experiment CrN film was deposited on a stainless steel substrate. Residual stress in the CrN film was measured by X-ray diffraction. The X-ray residual stress measurement revealed that macroscopic compressive stress and microscopic stress in the film are reduced by annealing the film/substrate system. The relation between the lattice strain and the applied stress is investigated for the specimen annealed at various temperature stage.

10:20am **TF-FrM7 Characterization of TiN Films Deposited on Electroformed Nickel Substrates**, ***M. Telgarsky***, *M.K. Ghantasala, E.C. Harvey, Y. Wang,* Swinburne University of Technology, Australia

The properties of thin films are mainly influenced by that of their substrates viz., the structure and microstructure. This paper describes the results of our investigations in understanding the effect of structure and microstructure of the electroformed Nickel substrate on the growth and properties of deposited TiN films. Nickel substrates were electroformed using two different Nickel sulfamate baths to thicknesses of between 100 and 200 μm. The grain size of these films was estimated to be around 15 to 50 nm. The grain size of the films formed in bath 1 was much larger than grains formed in bath 2. Interestingly, the films plated in bath 1 showed (200) orientation compared to those deposited in bath 2, which exhibited (111) orientation. Further, Nickel plated in bath 1 showed much better crystallinity compared to that deposited in bath 2. After releasing from their substrates, the electroformed Nickel foils were used in turn as substrates for the deposition of TiN thin films using filtered arc deposition (FAD). All films were deposited on the Nickel substrates under the same conditions. The electroformed Nickel and the TiN films were analyzed using Rutherford Backscattering Spectroscopy (RBS), X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM) and Confocal Microscopy for analysis of composition, structure, microstructure and surface profile respectively. The presence of small quantities of oxygen was detected in the electroformed Nickel from both baths. RBS analysis of the TiN films indicated that they were stoichiometric. TiN films deposited at room temperature were nano-crystalline, while those deposited at higher substrate temperatures showed the formation of improved crystalline structures having a (111) or (200) orientation. The grain size of the TiN films varied between 35 to 70 nm and showed a strong dependence on the grain size of Nickel and substrate temperature during deposition. The dependence of the TiN film properties on the Nickel substrate characteristics will be discussed in detail in this paper.

10:40am **TF-FrM8 Novel Mechanical, Electrical and Optical Properties of Al and Mg Doped Boron Thin Films**, ***Y. Tian***, Iowa State University

A new superhard and conductive (Al, Mg)-doped boron thin film with AlMgB@sub 14@ stoichiometry has been developed in this work for potential application as a hard coating on MEMS components and conductive atomic force microscope cantilevers. AlMgB@sub 14@ films were prepared on Si (100) using pulsed laser deposition at room temperature and 573 K. Transmission electron microscopy analysis reveals that the film structures are amorphous irrespective of substrate temperature during deposition. Nanoindentation tests show that hardnesses of 45 GPa and 51 GPa have been achieved in AlMgB@sub 14@ films deposited at room temperature and 573 K respectively. Except for their superhardness, AlMgB@sub 14@ films also display very low electrical resistivity and high optical absorption in a broad spectrum range, all these phenomena can be accounted for based on an electron transfer mechanism enhanced in amorphous structures.

11:00am **TF-FrM9 Synthesis of Super-Elastic Fullerene-Like Carbon Nitride Coatings by Unbalanced Reactive Magnetron Sputtering**, ***J. Neidhardt,*** Linköping University, Sweden*; Zs. Czigány,* Hungarian Academy of Sciences*; L. Hultman,* Linköping University, Sweden

Carbon nitride (CN@sub x@) is an emerging material for wear-resistant coatings. Out of several characteristic structures, the so-called fullerene-like (FL) compounds are the most promising. The FL structure leads to extraordinary mechanical properties such as an extreme elasticity combined with high fracture toughness as assessed by nano indentation. Yet, FL-CN@sub x@ shows a low-to-moderate resistance to penetration. Hence, deformation energy is predominantly stored elastically and released after unload giving it the resilient character. The key for understanding its resilient character lies in a unique microstructure of bent, cross-linked and frequently intersecting nitrogen-containing graphite sheets, denoted "fullerene-like". This presentation elaborates on the growth mechanisms of FL CN@sub x@ synthesised by unbalanced reactive magnetron sputtering of graphite in a nitrogen-containing atmosphere. Preformed C@sub x@N@sub y@ (x, y < 2) species originating from the target were assigned a crucial role for the fullerene-like structure evolution. In fact, the conducted plasma analysis revealed that the majority of the film forming flux comprises multi atomic nitrogen-containing species, which are partly sputtered and also thermally emitted from the self-modified target surface. The arrival of C@sub x@N@sub y@ molecules besides carbon atoms at the substrate surface adds much complexity to the growth mechanisms. C@sub x@N@sub y@ species may act as growth templates whereas an oriented incorporation along the edges makes them more stable towards preferential chemical desorption and therefore the formation of extended and curved sheet-like structures is promoted. Also the role and extent of temperature dependent chemical desorption (sputtering) process for the FL structure evolution was investigated in order to identify the type of species incorporated.

11:20am **TF-FrM10 Hydrogenated Carbon Films and Sulfur Stabilization: Synthesis and Tribiological Characterization**, ***C.A. Freyman,*** *Y.H. Yu, Y.W. Chung,* Northwestern University

Hydrogenated carbon films have been deposited on Si substrates and stainless steel ball bearings by magnetron sputtering. Hydrogen concentration was controlled by the percentage of hydrogen in the Ar/H@sub 2@ mixture. The friction coefficient showed a strong dependence on both the hydrogen content of the precursor gas and the relative humidity during testing. Twenty-five percent of hydrogen in the precursor gas produced the lowest frictional coefficient (0.01) at the lowest relative humidity measured (5%). Sulfur was incorporated into the film via the introduction of hydrogen sulphide in the precursor gas. Here, we attempted to use sulphur to make the surface hydrophobic and hence minimize the relative humidity effect. Hardness, friction and sensitivity towards the environment will be investigated as a function of deposition parameters.

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