Thin Films
Room 123 - Session TF-TuA

Growth and Properties of Thin Films

Moderator: P. Barna, Hungarian Academy of Sciences

2:00pm **TF-TuA1 Mechanical Properties and Stresses in Ion-Assisted Thin Films**, ***G.S. Was***, University of Michigan **INVITED**

Understanding the origin of stresses in thin films is critical to the control of film properties. Experiments were conducted to determine the origin of residual stresses in amorphous Al@sub2@O@sub3@ and crystalline Al and Nb films, and to control the stre sses in multilayer structures using ion-assisted deposition. Monolithic films were deposited during bombardment by Ne, Ar or Kr ions over a narrow range of energies, E, and a wide range of ion-to-atom arrival rate ratio, R. Films were characterized in terms of composition, thickness, density, crystallinity, microstructure, hardness and residual stress. Stress varied strongly with ion beam parameters, and with the resulting gas content. Residual stress and gas content saturated at a normalized energy of ~20 eV/atom or an R of ~0.05. Where residual stress varied linearly with RE@super1/2@, results are consistent with an atom peening model, but saturation at high R or RE@super1/2@ is inconsistent with such a model. The various mechanisms for residual stress control in monolithic amorphous and crystalline solids and the application of residual stress control to multilayered structures will be discussed. In Nb and Al films, the mechanisms for controlling crystallographic texture and the application of texture control for improved film properties are also presented. In both systems, texture developed slowly, but produced remarkable effects on plastic flow in the film. Specifically, examples will focus on crystallographic texture control in order to control the strength of the film/substrate interface and susceptibility to plastic flow in the film.

2:40pm **TF-TuA3 Growth of Highly-Oriented CeO@sub 2@ Layers on Glass Substrates for High-Quality Poly-Si Overlayer Formation**, ***N. Sakamoto***, *T. Inoue, T. Suzuki, S. Shida,* Iwaki Meisei University, Japan*; K. Kato,* Fukushima Technology Centre, Japan

In order to realize high-performance thin-film transistors, tremendous efforts have been made for growth of high quality polycrystalline Si (poly-Si) films on glass substrates. CeO@sub 2@ films on glass substrates have a potential advantage in attaining high-quality poly-Si overlayers without supplementary crystallization processes such as solid phase crystallization, excimer laser annealing and metal induced lateral crystallization. Systematic experiments varying growth temperature (room temperature 750°C) reveal that orientation controlled CeO@sub 2@ layers can be obtained. CeO@sub 2@ layers with strong (111)-tendency grow in low temperature region, whereas those with (100)-orientation grow at higher temperatures. Comparing conventional evaporation and electron-beam-assisted evaporation,@footnote 1@ the latter gives grain size enlargement and crystallization enhancement. For the films grown by electron-beam-assisted evaporation, the grain size estimated from the full width at half maximum of XRD peaks is 1.1-1.4 times larger than those grown by conventional evaporation. It is verified that poly-Si films with strong (111)-orientation are successfully formed on CeO@sub 2@/glass structures grown at room temperature. @FootnoteText@ @footnote 1@ T. Inoue, Y. Yamamoto, and M. Satoh, J. Vac. Sci. Technol. A, Vol. 19, Jan/Feb (2001) 275.

3:20pm **TF-TuA5 Thin Film Growth of Reactive Sputter Deposited Tungsten-Carbon Thin Films**, *P.D. Rack,* Rochester Institute of Technology*;* ***J.J. Peterson***, *J. Li,* Advanced Vision Technologies*; H.J. Rack, A.C. Geiculescu,* Clemson University

Tungsten-carbon thin films have been reactively sputter deposited in various Ar-CH gas mixtures and the growth kinetics of the reactive deposition process have been elucidated. X-ray diffraction data reveal that the films are amorphous as-deposited with partial crystallization of W2C and WC occurring following a 1100oC-1 minute rapid thermal anneal. Peak shape analysis of the W and C x-ray photoelectron peaks show binding energy shifts consistent with carbide formation for the annealed films. Carbon incorporation within the W-C films is attributed to flux of CH3 radicals impinging on the growth surface. Although the CH3 radicals have a significantly lower concentration (~0.1%) than the CH4 molecules contained within the plasma, the sticking coefficient of CH3 is significantly larger than that of CH4. In addition, the change in the incorporation rate of carbon in the W-C films at higher CH4 (and subsequently CH3) concentrations has been shown to be due to the changes in the growth surface; as the CH3 flux increases, the growth surface becomes carbon terminated decreases the incorporation of carbon because of the low CH3-C sticking coefficient. This presentation will demonstrate the experimental procedure used in growing the W-C thin films. Compositional analysis as a function of the CH4 concentration will be presented and the growth process will be shown to follow Langmuir-type growth.

3:40pm **TF-TuA6 Reactive Sputter Deposition of Tungsten Nitride Thin Films**, ***C. Baker***, *S.I. Shah,* University of Delaware

Tungsten nitride (WN@sub x@) thin films were deposited by reactive sputtering in an Ar/N@sub 2@ atmosphere. The partial pressure of nitrogen in the sputtering gas was varied from 2-50% and the effect of the N@sub 2@ concentration variation on the film properties was investigated. Through examination of cathode current and voltage during the film growth, it is determined that cathode poisoning occurs when the nitrogen concentration in the chamber is increased above 2-5%. This poisoning reduces the film growth rate. Films were characterized by X-ray Photoelectron Spectroscopy. XPS analyses show that the films are composed of ~30% nitrogen when the nitrogen concentration in the chamber is greater than 10%. X-ray diffraction analyses confirm that the predominant phase in the reactively sputtered films is W@sub 2@N, with the characteristic (111) peak found near 2@theta@=37.7Â°. Slight shifts in this peak position are thought to be the result of nitrogen incorporation in interstitial positions, thus distorting the lattice. A post-deposition anneal was carried out which shifted the peak back to its characteristic position confirming that the extra nitrogen was indeed accommodated as interstitial.

4:00pm **TF-TuA7 The Impact of Residual By-Products from Tungsten Film Deposition on Process Integration due to Non-Uniformity of the Tungsten Film**, ***A. Sidhwa***, STMicroelectronics, Inc. and University of Arkansas*; C. Spinner, T. Gandy, S. Melosky,* STMicroelectronics, Inc.*; W. Brown, S. Ang, H. Naseem, R. Ulrich,* University of Arkansas

The effects of residual by-products from a tungsten film deposition process and their impact on process integration due to the non-uniformity of the tungsten film were investigated in this work. The tungsten film deposition process involves three steps: nucleation, stabilization, and tungsten bulk fill. Six experiments were conducted in search for a solution to the problem. The resulting data suggest that excess nitrogen left in the chamber following the tungsten nucleation step, along with residual by-products, causes a shift in the tungsten film uniformity during the tungsten bulk fill process. Data reveal that, due to the residual by-products, an abnormal grain growth occurs causing a variation in the tungsten thickness across the wafer during the bulk fill step. Although several possible solutions were revealed by the experiments, potential integration problems limited the acceptable solutions to one. The solution chosen was the introduction of a 10 second pumpdown immediately following the nucleation step. This choice did not create any integration problems as confirmed by subsequent studies.

4:20pm **TF-TuA8 Thermal Stability of Arc Evaporated Ti@sub 1-x@Al@sub x@N Thin Films**, ***A. Hörling***, *L. Hultman, M. Odén,* Linköping University, Sweden*; G. Ramanath,* Rensselaer Polytechnic Institute*; P.H. Mayrhofer, C. Mitterer,* University of Leoben, Austria*; J. Sjölén, L. Karlsson,* Seco Tools, Sweden

The thermal stability of Ti@sub 1-x@Al@sub x@N thin films deposited by arc evaporation from cathodes with nominal composition x=0.67 onto substrates kept at 500°C has been investigated by XRD and TEM, and by differential scanning calorimetry (DSC) and 4-point probe measurements. As-deposited films contained 62 at.% Al and were of cubic [NaCl]-structure phase. Annealing of such metastable films results in relaxation of intrinsic compressive stress, together with spinodal decomposition into cubic TiN and AlN-rich phases succeeded by the precipitation of hexagonal AlN. Stress relaxation was observed to take place for all annealing temperatures above the deposition temperature, which implies that point defects or defect complexes become annihilated. For example, DSC showed enthalpy changes at temperatures up to 700°C, and XRD showed a decrease in peak broadening at temperatures up to 800°C. By XRD, the spinodal decomposition stage was revealed as a symmetrical broadening of the cubic (Ti,Al)N (200) peak after annealing at 900°C; by DSC as an exothermic peak starting at 850°C for a heating rate of 27°C min@super -1@; and by 4-point probe measurements as an increased sheet resistance at 500°C with a maximum at 650°C for a heating rate of 5°C min@super -1@. At higher temperatures, the resistivity decreased, and upon cooling and re-annealing showed a reversible behaviour which indicates a parallel-circuit behaviour of a more TiN-like matrix together with emerging AlN phases. Following

spinodal decomposition, phase separation of the structure into c-TiN and h-AlN occurred in the temperature range of 900°C-1100°C, being observed by XRD and TEM. The 4-point probe measurements, especially when compared to XRD, indicate that the heating rate has a large effect on the kinetics of phase separation. This observation will be discussed, together with results of activation energies for the various reactions.

4:40pm **TF-TuA9 Evolution of Ti-3Al Film Structures and its Effect on Film Properties**, ***C.-F. Lo***, *D. Draper, P. McDonald, P. Gilman,* Praxair-MRC

The evolution of Ti-Alx film structure deposited from the Ti-75at%Al (Ti-3Al) sputter targets was investigated. 300nm, 900nm and 9000nm thick films were deposited on three inch diameter silicon wafers at various wafer temperatures from 20°C to 400°C. The films were evaluated for composition, microstructure, crystallinity, hardness and elastic modulus using the FE-SEM, XRD and nano-indentation instrument. In order to understand the effect of the target structure on film properties, the target manufacturing process for the Ti-75 at%Al (Ti-3Al) alloy system was controlled to prepared two four inch diameter targets, one metallic (Ti+3Al) and one intermetallic (TiAl@sub 3@) structures. No effect of target structure and sputtering conditions on the composition of the deposited films was observed. The film compositions were similar to that of the sputtered targets. At a film thickness 300nm and less, the grains showed an equiaxial shape with size about 40nm. The columnar grains generated and grew with increasing of film thickness. The diameter of the columnar grains increased with increasing of film thickness. The target structure showed some effect on the morphology but not on the diameter of columnar grains. XRD analysis showed that TiAl was the major phase existing in all the tested films. The hardness and elastic modulus measurements showed that the mechanical properties of the deposited films were effected by the film microstructure.

5:00pm **TF-TuA10 Surface Structural Anisotropy in Sputter and Electrolytic Deposited Tantalum Films**, ***S.L. Lee***, US Army Armament Research Development and Engineering Center*; D. Windover,* Rensselaer Polytechnic Institute

We studied the growth surface texture of tantalum films generated by sputter and electrolytic deposition processes for high temperature and pressure wear and erosion applications, using conventional and image plate XRD. Electrochemically deposited bcc tantalum coating in eutectic molten salt solution at 800°C on 20mm-diameter 4340 steel cylinder revealed near random grain orientation. Triode sputtered tantalum deposition on steel generally showed weak anisotropy. Example specimens deposited in krypton gas at 200°C-250°C revealed (110) and (211) texture with poles. Planar magnetron sputtered tantalum film on a steel plate with a thin electrolytic chromium interface layer produced bcc tantalum film with (111) fiber texture, following the (111) fiber texture in chromium. Planar magnetron sputter-deposited 200nm film on a (100) silicon wafer, mixed bcc and tetragonal tantalum and (110) fiber texture with azimuth symmetry evolved. Random oriented grain distribution suggests uniform thermal-mechanical properties in polycrystalline solids. Model calculations of Young's modulus and Poisson's ratio for isotropic tantalum crystalline aggregates were made. Evaluation of directional modulus, e.g., E110, E100, E111, from single crystal elastic constants, suggested higher elastic modulus in the out-of-plane direction for (111) fiber-textured films.

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