Thin Films Division
Room 615 - Session TF-TuA

Fundamentals of Si and Dielectric PVD

Moderator: S. Zarrabian, Optical Coating Labs

2:00pm **TF-TuA1 Preparation of Co and CoN@sub x@ Thin Films by Unbalanced r.f. Magnetron Sputtering**, ***T. Tanaka,*** Hiroshima Institute of Technology, Japan; *A. Kitabatake,* Sanyo Shinku Kogyo, Japan; *K. Kawabata,* Hiroshima Institute of Technology, Japan

It has been difficult to deposit ferromagnetic thin films by using a conventional planar magnetron sputtering at low pressure. We have developed a modified process based on an unbalanced magnetron sputtering of a magnetic Co target (100 mm, 5mm thick) to deposit Co and CoN@sub x@ films where an external magnet is added to a conventional planar magnetron to confine the efficient plasma near the magnetron target. The plasma confinement can be controlled by the shape of the magnetic field in the sputter deposition device with a multipolar magnetic-field plasma confinement. Cobalt films were prepared by this sputtering system at the r.f. powers of 100 to 200 W and argon pressure less than 5x10@super -3@ Torr. It is shown that the deposition rate of Co films significantly increases from 8.7 to 25 nm/min whose values are two times that of a conventional magnetron sputtering. Co film with the preferred orientation of (111) plane is formed and the value of the grain size estimated from the plane is about 30 nm. Cobalt nitride (CoN@sub x@) films were also prepared by the unbalanced magnetron sputtering in mixtured argon and nitrogen plasma. It is also found from the results of electron probe microanalysis that the content of nitrogen in CoN@sub x@ films increases with the increasing gas flow ratio of N@sub 2@ . The electrical resistivity for reactively sputtered films is less than 80x10@super -6@ @ohm@ cm which makes this compound a relatively good conductor.

2:20pm **TF-TuA2 Microstructural Control of Thin Silicon Films Grown by Reactive Magnetron Sputtering Utilizing Low Energy Ion Bombardment**, ***J. Gerbi,*** *J.R. Abelson,* University of Illinois, Urbana-Champaign

We use spectroscopic ellipsometry, Raman scattering, TEM, SIMS, and photoluminescence to analyze the optical properties and microstructure of hydrogenated or deuterated Si thin films of various crystallinities. Bulk mc-Si films are of current interest for solar cell, hybrid solar cell, and thin film transistor applications; low temperature deposition enables the use of plastic substrates. We have previously demonstrated that RMS can deposit mc-Si films directly on glass with no amorphous boundary layer,@footnote1@ and that substituting D2 for H2 in the growth process enhances crystallinity.@footnote2@ In this work, we deposit 0.5 micron thick hydrogenated or deuterated mc-Si films on glass substrates by RMS of a Si target using 1.6 mT Ar plus H2 or D2 at partial pressures from 0 mT (producing amorphous films) to 5.5 mT (producing fully microcrystalline films) at substrate temperatures of 120 and 230 C. In our system, the ion flux and energy are decoupled parameters. The ion flux is controlled by the application of a cylindrical magnetic field created by external coils. This field directs a weak plasma toward or away from the substrate, controlling the ion flux such that the ratio of arriving ions to depositing Si atoms can be varied from < 1 to > 30. The ion energy is also externally controlled by biasing the substrate. We find marked differences in microstructure using high ions fluxes at energies <~ 30 eV, and we will report both the microstructural and optical properties of the films. We also show that biasing of the substrate to produce ion energies >= 50 eV (as often done in conventional diode sputtering systems at higher pressures) produces damage which degrades the mc-Si microstructure. @FootnoteText@ @footnote 1@Y. H. Yang and John R. Abelson, Appl. Phys. Lett. 67, 3623 (1995). @footnote 2@ J. E. Gerbi and John R. Abelson, "Enhanced Crystallinity of Microcrystalline Silicon using Deuterium in Low Temperature Reactive Magnetron Sputter Deposition," MRS Proc. 507, 429 (1998).

2:40pm **TF-TuA3 PVD of Thin Film Silicon: How Fast Light Atom and Slow Heavy Ion Bombardment During Growth Promote Low-Temperature Crystallinity**, ***J.R. Abelson,*** University of Illinois, Urbana **INVITED**

Macro-electronic devices such as photovoltaic cells and active matrix displays are based on the deposition of thin semiconductor films onto large area substrates at low temperatures. Silicon presents an interesting case because the microstructure can range from amorphous to nanocrystalline to polycrystalline. These different microstructures can be produced by manipulating the concurrent particle bombardment during PVD growth by dc reactive magnetron sputtering of a Si target in Ar + H2. Three types of particles impinge on the film: (i) sputtered Si atoms of a few eV; (ii) H atoms with ~ 100 eV, generated by the acceleration and reflection of H2+ ions at the target; and (iii) bulk plasma Ar+ and H2+ ions with ~ 25 eV, whose flux is controlled using an externally-generated magnetic field to unbalance the magnetron. We analyze the growth process using real-time mass spectroscopy, spectroscopic ellipsometry, and reflection IR absorption, including isotopic H2/D2 exchange experiments. We combine these data with binary collision simulations in the gas-phase and substrate to show how each flux modifies the microstructure. The essential results are: (i) Few-eV sputtered Si atoms produce a dense microstructure, as predicted by the Thornton zone diagram, but also lead to the random formation of nanocrystalline Si particles in an amorphous Si matrix. These particles can serve as nuclei for solid-phase crystallization processes. (ii) 100 eV H atoms penetrate ~ 50 A into the growing film, where they drive crystalline nucleation and subsurface transformation through bond-insertion and momentum-transfer events. Fully nanocrystalline films can be deposited on glass substrates using large fluxes of fast H or D atoms. (iii) 25 eV Ar+ ions modify the competitive growth of polycrystalline grains at the film surface, which leads to a coarsening of the grain structure at modest substrate temperatures.

3:20pm **TF-TuA5 Deposition Behavior and Film Characteristics of Aluminum Oxide Deposited using High Frequency Pulsed-DC Magnetron Reactive Sputtering**, ***D.C. Carter,*** *G.W. McDonough, L.J. Mahoney, G.A. Roche, H.V. Walde,* Advanced Energy Industries

The affects of pulsed-DC power application in reactive magnetron sputtering of insulating films has been the subject of much study in recent years. Improved process stability with decreased arcing incidence and cleaner films have resulted by applying bi-polar pulsed power at frequencies from 10 to 200 kHz to otherwise traditional DC magnetron reactive sputtering processes. Recent advances in power supply design, however have extended the usable range of DC pulsing to 300 kHz and above. At these extended frequencies it is observed that transient behavior in the magnetron discharge becomes increasingly dominant on the measured waveforms of the applied power. Little is known of how this behavior affects the dynamics in a reactive sputtering environment or how these high pulsing frequencies can act to influence the character of films reactively deposited. This study looks specifically at the affect high frequency DC pulsing has on reactively sputtered aluminum oxide. Target voltage and partial pressure hysteresis behavior are reviewed from 0 to 350 kHz to ascertain the affect pulsing frequency has on sputter target condition. Deposition rate and film properties of hardness and optical transmission are reviewed to better understand the impact high frequency pulsing has on the deposited material itself.

3:40pm **TF-TuA6 AC Reactive Sputtering of Dielectric Films using a Dual Magnetron**, ***A. Belkind,*** *J. Cai,* Stevens Institute of Technology; *R. Scholl,* Advanced Energy Industries, Inc.

DC reactive sputtering to produce dielectric films suffers from two problems: Arcing on the target surface and covering the anode (the 'disappearing anode problem'). Both problems have received serious attention in recent times. One way to solve both simultaneously is to apply ac power between two magnetrons. Although this approach was first suggested more than ten years ago, and has been widely implemented, a detail investigation of it is remains lacking. In this work, ac reactive sputtering from a dual magnetron system is studied. The effects of ac frequency and discharge current on reactive sputtering of aluminum oxide using both balanced and unbalanced magnetrons are investigated. Special attention is given to ion bombardment of a substrate, both electrically floating and connected to the power supply system.

4:00pm **TF-TuA7 Characterization Studies of Reactively Pulsed Magnetron Sputtered Alumina Films**, ***P.J. Kelly,*** *P.S. Henderson, R.D. Arnell,* University of Salford, UK

It is well-established that pulsing the magnetron discharge during the reactive sputtering of insulating films, particularly alumina, can significantly reduce arc events at the target. The suppresion of arc events stabilises the reactive depositon process and, thus allows control over the coating composition, structure and properties. Fully dense, defect-free ceramic films can now be routinely produced at high depositon rates using the pulsed magnetron sputtering process. However, despite the success of this process, optimum deposition conditions and the relationships between deposition conditions and film properties are not well reported. In this investigation, alumina films have, therefore, been deposited by reactive magnetron sputtering using various combinations of DC and pulsed DC power. The deposition conditions, including pulse frequency, reverse voltage and reverse time, were systematically varied, and the coatings were characterised in terms of their structures and properties. Properties measured include nanohardness, resistivity and scratch adhesion. The optical properties of the coatings have also been investigated. In addition, the power supplies and pulse units were characterised in terms of their effectiveness at arc suppression and their suitability for particular deposition processes. A range of operating conditions have been identified over which hard arcs are fully suppressed and coatings with consistent properties are produced.

4:20pm **TF-TuA8 Change in Surface Roughness with the Thickness of TiO@sub2@ Film Grown on MgO(001) by Ar-ion Beam Sputtering***, T. Uchitani, **K. Maki**,* Yokohama City University, Japan

Thin film growth mode is closely correlated with the surface roughness as predicted from some simulation by Kim and Kosterlitz (Phys. Rev.Lett., vol. 62, 2289(1989)). According to their study, the surface roughness, R@sub a@, is proportional to d@super L@ with L = 1/(D+1), where d and L represent the film thickness and the dimension, respectively. The rutile-type TiO@sub 2@ film was deposited on air-cleaved MgO(001) held at 630 °C at 3.1 x 10@super -3@ Pa in the partial pressure of O@sub 2@ and at 7.9 x 10@super -3@ Pa in the pressure of Ar by sputtering the Ti target by Ar-ion beams accelerated at 1.2 kV. The R@sub a@ versus d relationship at d > 10 nm was determined with an AFM observation, and the film crystallinity was evaluated by determining the relationship between the intensity ratio of (110) peak of TiO@sub 2@ to (004) peak of MgO in X-rays diffraction pattern and d@super 2@. By determining the former relationship, the amount of L is estimated to be 1/2 and so D is one. In other words, the growth of TiO@sub 2@ film at d > 10 nm in the present study progresses by atom by atom process which is not accompanied with the surface diffusion for the adatoms and some atomic rearrangement in the condensed phase during the film deposition. This means that the film crystallinity is independent of d which is supported from the linear relationship between X-rays diffraction intensity peak ratio and d@super 2@.

4:40pm **TF-TuA9 Chemical Vapor Deposition of Alpha Aluminum Oxide for High Temperature Aerospace Sensors***, **R.H. Niska**,* AlliedSignal Aerospace Co.*; A.P. Constant, T. Witt,* Iowa State University*; O.J. Gregory,* University of Rhode Island

Thin film thermocouples and strain gages are being developed for high temperature application on aerospace propulsion hardware for both development test purposes and as active control sensors. The critical technology necessary in the fabrication of the sensor is an adherent, dense, and homogeneous dielectric to provide electrical isolation at engine operating temperatures. Techniques are being developed to create a crystalline aluminum oxide dielectric formed by a combination of a thermally grown oxide [TGO] from a NiCoCrAlY hardcoating which is then enhanced with the addition of a chemical vapor deposited [CVD] crystalline aluminum oxide layer. This paper will focus on the process development used to deposit the alpha alumina layer on the TGO using CVD in a coldwall reactor at 1100C. The chemistry employed in this process is the pyrolitic decomposition of aluminum tri-isopropoxide. The hexagonal [HCP] alpha phase is achieved at deposition temperatures of 1000C-1100C, as confirmed by X-ray diffraction analysis. By eliminating gas phase and hot wall decomposition, this approach minimizes precursor depletion effects, yielding a more dense and uniform film morphology. Conformal coatings up to 10 microns thick with high resistivity and good adhesion and hardness have been observed on complex airfoil geometries. Growth rates up to 10 microns per hour are possible although low growth rates lead to more desirable film properties. The kinetics of the deposition indicate that the reaction proceeds by a mass transport limited mechanism. Uniform temperature control over highly complex geometry is desireable, but not essential for uniform film growth. Results indicate that the gas flow uniformity and the precursor transport rate are the critical variables.

5:00pm **TF-TuA10 Phase Development of Radio Frequency Magnetron Sputter Deposited Pb(Mg@sub 1/3@Nb@sub 2/3@)O@sub 3@-PbTiO@sub 3@ (90/10) Thin Films***, **J.-K. Lee**,* Korea Institute of Science and Technology, Korea*; D.K. Park, D.-S. Cheong,* Korea Institute of Science and Technology*; J.-W. Park,* Hanyang University, Korea

The electrostrictive properties of relaxors, PMN-PT, have been the focus of intensive studies in view of their application in microactuator. This is because at around the dielectric constant maximum, relaxors exhibit large electrostrictive strain, the strain-field relationship is practically free of hysteresis, and the effective piezoelectric coefficient can be tuned by changing the magnitude of the dc bias field. PMN-PT films were deposited by r.f. magnetron sputter deposition from Pb, Mg enriched ceramic targets. The Perovskite structural analysis was confirmed by X-ray diffraction. Film growth was carried out over a wide range of processing parameters such as substrate temperature, sputtering pressures, and post annealing conditions. We focus on the formation of 100% Perovskite structured PMN-PT film with good electrostrictive properties. Composition and phase development were controlled by observing the sputter physics and the depositon mechanism. In case of the film containing the volatile species such as Pb and Mg, the sputtering pressure must be controlled. We also discuss the role of excessive MgO phase on the nanocomposite characteristics in PMN-PT films.

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