Vacuum Metallurgy Division
Room 328 - Session VM-MoA

Plasma Assisted Surface Treatments and Coatings

Moderator: I. Petrov, University of Illinois, Urbana

2:00pm **VM-MoA1 Low Temperature Growth of Protective Coatings in an ECR Plasma***, C.-T. Lin, F. Li,* ***T.D. Mantei,*** University of Cincinnati

A high density microwave electron cyclotron resonance (ECR) discharge has been used to grow hard, colorless, and transparent silicon dioxide barrier coatings at deposition rates up to 0.7 µm/min and substrate temperatures from 80 to 120°C. The deposition precursors tested were tetraethoxysilane (TEOS), hexamethyldisiloxane (HMDSO), and hexamethylcyclotrisiloxane (HMCTSO). Metal substrates were introduced into the process chamber through a vacuum loadlock and subjected to an in situ three minute argon plasma cleaning cycle. Oxygen was then metered into the chamber through ports located upstream just below the input microwave window, while the precursor reactant gas was introduced downstream. The total gas pressure prior to plasma ignition was 1 to 10 mTorr and the O@sub 2@/precursor flow ratios were varied from 2:1 to 8:1. The substrate temperature, measured with a clamped thermocouple, rose to 80-100°C within a few minutes and then rose slowly during the remainder of the deposition cycle to a final temperature less than 120°C. Final film thicknesses were 3 to 10 µm. Maximum deposition growth rates were 0.25 µm/min for TEOS, 0.65 µm/min for HMDSO, and 0.68 µm/min for HMCTSO, increasing with increasing precursor gas flow, increasing input power, and decreasing O@sub 2@/precursor flow ratio. Fourier transform infrared spectroscopy analysis showed mainly Si-O elemental bonding for all films, with small Si-CH@sub 3@ and SiOH components. Coating compositional analyses performed with X-ray photoelectron spectroscopy showed oxygen-to-silicon ratios of approximately 2:1, but with significant carbon percentages; e.g. the carbon fraction for TEOS films ranged from 29% with an O@sub 2@/precursor flow ratio of 2.5, down to 14% for an 8:1 flow ratio.

2:20pm **VM-MoA2 Low Temperature Polycrystalline Silicon Resistors on Glass Substrates***,* ***A.T. Krishnan****, S.H. Bae, S.J. Fonash,* Pennsylvania State University

Polycrystalline silicon (poly-Si) thin film resistors have been processed on glass substrates at low temperatures (<300@degree@C) using a novel approach. This approach involves direct deposition of n+ polycrystalline silicon thin films using an electron cyclotron resonance (ECR) high density plasma (HDP) tool, with phosphine as the dopant source. These n+ poly-Si films have been deposited on both Corning 1737 glass and soda-lime glass substrates at 300@degree@C. The dependence of film quality on film thickness has been exploited to obtain devices with resistivities over a range of 1-10 ohm-cm. The range of resistivities that can be obtained can be further increased by altering the phosphine flow rate. The deposition rates are of the order of 100 Å per minute. The temperature coefficient of resistivity of these devices is less than 2000 ppm/@degree@C. Preliminary studies indicate that it is possible to obtain n+ poly-Si films at temperatures lower than 200@degree@C, which would allow the use of clear flexible plastic substrates. Thin film resistors processed at low temperatures find a wide range of applications, such as flat panel displays, multi-chip modules,@footnote 1@ anolog circuits, @footnote 2@ and high frequency applications.@footnote 3@ In CMOS technology, low temperature resistor fabrication would enable integration of resistors on fully processed Si chips. Conventional non silicon based resistor technology requires more than one material, such as a cermet for high resistivities and a metal for low resistivities, to achieve a wide resistivity range. Currently existing Si based resistor technologies, like LPCVD cannot be used for low temperature applications, because of the high deposition temperatures (600@degree@C, which is close to aluminum melting point) or higher temperatures needed for dopant activation (700-800@degree@C). The HDP technique allows the use of a single material (n doped poly-Si) to obtain a wide range of resistivities. Because it is Si based, it is compatible with current ULSI processing techniques. Its low deposition temperature and the fact that no annealing is required to activate dopants makes it ideally suited for low temperature applications listed above. @FootnoteText@ @footnote 1@Integration of passive components for microwave filters in MCM-D, Pieters, Philip; Brebels, Steven; Beyne, Eric, Proceedings of the 1997 6th International Conference and Exhibition on Multichip Modules 1997 Denver, CO, USA p 357-362. @footnote 2@BiCMOS analog front-end circuit for an FDM-based ADSL system Langford, D. Scott; Tesch, Bruce J.; Williams, Brian E.; Nelson, G. Rodney; Ross, Robert B.; Bechtel, Gerry R.; Lewis, Mike G, Proceedings of the 1997 Bipolar/BiCMOS Circuits and Technology p 180-182. @footnote 3@110 GHz slow-wave electrodes for velocity-matched distributed MSM photodetectors with integrated bias load, Pfitzenmaier, H.; Boettcher, E.H.; Droege, E.; Bimberg, D, Proceedings of the 1997 IEEE Lasers and Electro-Optics Society Annual Meeting, USA p 218-219

2:40pm **VM-MoA3 Closed Loop Control of Reactive Sputtering of Oxide Thin Films***,* ***L. Lou****, M. Mai, G.W. McDonough, H.V. Walde, R. Scholl, G.A. Roche,* Advanced Energy

Rapid closed loop control of oxygen flow was used to prevent target poisoning and facilitate high growth rate during reactive sputtering. Low frequency AC reactive sputtering of Al@sub 2@ O@sub 3@ and SiO@sub 2@ was performed achieving stable film formation with high deposition rates. Thin film properties of rate, refractive index and stress are reported as a function of pressure and power. Results are compared to reactive sputtering of these films by other techniques.

3:00pm **VM-MoA4 Unbalanced Magnetron Sputtered Composite Metal-DLC Coatings***,* ***X.T. Zeng****,* Gintic Institute of Manufacturing Technology, Singapore

Composite metal-diamond like carbon (DLC) coating is an approach to achieve a combination of high hardness and toughness, good adhesion, a low friction coefficient, and a low wear rate, which is ideal for various tribological applications. Previous studies often used hydrocarbon gases to produce amorphous a:C-H coating doped with metal (Ti, W, Cr) and/or their nitrides by magnetron sputtering from metallic targets. Relatively high hardness (15 ~ 20 GPa) could be obtained in these coatings by increasing the doping level at the expense of an increasing friction coefficient which results in high wear rates. This paper reports the preparations of composite metal-DLC coatings on high speed steel substrates using unbalanced magnetron sputtering of both metallic and graphite targets. W and Ti/Cr targets were used to deposit both the bond layer and transition ceramic layer to support the surface composite DLC coating. For comparison, amorphous CN@sub x@ surface coating was also prepared for sliding wear testing. Nanoindentation and scratch tests were used to characterize the mechanical properties and pin-on-disk wear tests, using @phi@9 mm alumina as the pin and 10 N normal load, were carried out to evaluate the tribological properties of the coatings. Friction coefficient of about 0.06 ~ 0.1, at an air humidity of 40% RH, hardness of 15 ~ 18 GPa, critical load of about 65 N in scratch tests, and normalized wear rate of about 5 x 10@super -8@ mm@super 3@.N@super -1@.m@super -1@ for wearing for 38000 to 76000 cycles (3400 m to 6800 m) were measured. This wear rate is substantially lower than those of the nitride coatings. The superior wear resistance coupled with good adhesion implies that composite metal-DLC coatings could be good candidates for sliding wear applications.

3:20pm **VM-MoA5 Overview of Plasma Source Ion Implantation***,* ***J.R. Conrad,*** University of Wisconsin, Madison **INVITED**

Plasma Source Ion Implantation (PSII) represents a radical departure from conventional ion implantation technology. PSII circumvents the line of sight restriction inherent in conventional ion implantation. In PSII, targets to be implanted are placed directly in a plasma source and then pulse-biased to a high negative potential. A plasma ion matrix sheath forms around the target and ions bombard the entire target. Compared with conventional ion implantation, PSII minimizes the problems of shadowing and excessive sputtering of the target material, which can severely limit the retained dose of the implanted ion species. This talk will present: a historical overview of the development of PSII; a brief review of PSII physics and technology; a summary of world-wide PSII activities; a discussion of laboratory and industrial field test results in PSII; recent activities leading to scale-up and commercialization of PSII; recent extensions of PSII technology to semiconductor processing.

4:00pm **VM-MoA7 Improvement of Tribological Properties of Pure Aluminium by Isotropic ECR Ion Implantation***,* ***D. Popovici****, B. Terreault, A. Sarkissian, B.L. Stansfield, R.W. Paynter, G.G. Ross,* INRS-Energie et Materiaux, Canada

There are serious limitations to using Al and its alloys for light-weight components: in the absence of lubrification they have relatively poor tribological properties such as high friction and wear rates in sliding contact. These characteristics are due to a low flow stress of the metal and the brittleness of the aluminum oxide. The improvement of the tribological characteristics of Al, by conventional and high energy(@>=@50keV)nitrogen and oxygen implantation has already been demonstrated. In the case of N@super +@ implantation the increase in hardness is due to the formation of an AlN film. Because relatively high substrate temperatures(400-500°C)are necessary when conventional plasma nitriding is used, the difference in the thermal expansion coefficients of Al and AlN leads to microcracks in the AlN coatings. In the case of O@super +@ implantation, the formation of a maetstable phase is followed by a thermal annealing, inducing the synthesis of nanoprecipitates of Al@sub 2@O@sub 3@ which are dense enough to pin all dislocations. In this study, we used a low temperature, low energy (30keV)isotropic PBII whith an ECR plasma source, to investigate and compare near-surface N@super +@ and O@super +@ implantation into high purity (99,99%) Al. The surface chemical composition and chemical bond formation of the implanted Al layer were investigated by AES, XPS and RBS. The surface tribological properties, structure and cristallinity were analysed by lateral force microscopy (LFM), nanoindentation and XRD. Implantation depth profiles were determined for several implantation times and sample-plasma geometries. The profiles were found to agree with TRIM simulations that supposed a monoenergetic ion source. The implantation of monoenergetic ions by our PBII technique, using a high frequency pulsed plasma and a steady high voltage for ion acceleration, allows for a precise tailoring of the implantation depth profile.

4:20pm **VM-MoA8 Molecular Dynamics Study of Al PVD Processes***, **U.P. Hansen**, P. Vogl,* Technical University Munich, Germany*; A. Kersch,* Siemens Ag, Germany

We present a computationally efficient classical many body potential that has been designed to model the Al-Al interaction in a wide range of bonding geometries ranging from bulk AL to Al surfaces and to the Al@sub 2@ dimer. It is shown that this potential yields Al elastic constants, Al surface diffusion barriers, surface formation energies and Al@sub 2@ properties in excellent agreement with experiment and/or previous ab-initio results. Detailed molecular dynamics simulations are performed that elucidate the different surface reactions taking place during Al physical vapor deposition. We find a high sticking probability for Al atoms impinging normal on Al surfaces and the sticking coefficient is decreasing with increasing incident angle to the surface normal. Detailed explanation for this prediction including atomistic surface reactions is presented. The energy dependence of the sticking probability calculated by our model deviates clearly from predictions of a simple hard sphere picture.

4:40pm **VM-MoA9 An XPS Study of the Effects of Chemical Pre-Cleaning of Aluminum Alloys on the Anti-Corrosion Properties of Plasma Deposited Films***, **C.E. Moffitt**, D.M. Wieliczka,* University of Missouri, Kansas City*; H.K. Yasuda,* University of Missouri, Columbia

DC-plasma deposited films are showing great promise as an alternative to chromate conversion for enhanced coating adhesion and corrosion protection of the aircraft alloys AA2024 and AA7075. The oxide structure of these alloys is usually modified by chemical treatments prior to application of corrosion resistant coatings. The effects of cleaning with certain chemistries employed in industry were investigated with XPS sputter depth profiling. This study of the remaining oxide layer indicates that the cleaning process leaves some undesirable deposits on the surface, which affect the stability of the coating/alloy interface region during corrosive attack. The XPS depth profiles reveal the extent of the changes in alloy surface composition after cleaning. Depth profiles of plasma film coatings on alloy surfaces treated with specific chemistries are also presented. This work was supported under Air Force contract AF F33615-96-C-5055.

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