Applied Surface Science
Room 206 - Session AS+TF-ThA

Thin Film Characterization II
Moderator: K. Lloyd, Dupont

2:00pm **AS+TF-ThA1 C@sub n@ (50@<=@n<60) Films on HOPG**, ***A. Böttcher***, *P. Weis, S.-S. Jester, D. Löffler, M.M. Kappes,* Universität Karlsruhe, Germany

Novel solid materials have been grown under ultra high vacuum conditions by gentle deposition of C@sub n@@super +@ (50@<=@n<60) on HOPG surfaces (kinetic energy typically < 0.1 eV/atom). C@sub n@@super +@ ions resulting from the electron-impact induced ionization/dissociation of C@sub 60@ are driven by a system of electrostatic lenses through a mass spectrometer towards the HOPG substrate where an appropriate retarding potential assures their soft-landing. This setup enables to achieve deposition rates in the range of 10@super 12@ ion/s. AFM images reveal that the C@sub n@ films grow according to the Volmer-Weber scenario, i.e. the surface is initially dominated by 2D fractal islands, which in later deposition stages become 3D dendritic pyramids. This behavior stems from the aggregation of C@sub n@ cages, which is driven by reactive sites as formed by adjacent pentagons (or heptagons) on individual cages. The resulting covalent bonds are responsible for the unusually high thermal stability of the C@sub n@ films. Thermal desorption spectra of C@sub 58@ reveal activation energies around 2.2 eV, which are considerably higher than the sublimation enthalpy of C@sub 60@ films. AFM images taken after completing the desorption exhibit a network of highly polymerized cages, which remains stable even at temperatures around 1300 K. Recent DFT calculations as well as UPS-measurements support the aggregation scenario of C@sub n@ cages. Well recognizable features in the valence-band spectra allow identification of the band gap as well as contributions from C@sub n@-C@sub n@ bonds.

2:20pm **AS+TF-ThA2 An in situ Study on Amorphous Carbon Films and the Vapor Phase Lubrication in Magnetic Data Storage Media**, ***Y. Yun,*** *A.J. Gellman,* Carnegie Mellon University

Amorphous carbon films have been used as a protection for the magnetic layer in hard disks for years. Perfluoropolyalkylether (PFPE) has been applied on the amorphous carbon film surface as lubricant during the crashing of the read-write head on the amorphous carbon film. The fundamental understanding of the thermal stability and oxidation kinetics of amorphous carbon films and the adsorption of PFPE lubricants is essential to obtain the ultimate high performance protection and lubrication. Vapor phase lubrication integrates the amorphous carbon film deposition and lubrication in vacuum. The amorphous carbon film can be oxidized under controlled conditions immediately prior to lubricant adsorption. The kinetics of oxidation has been studied using x-ray photoelectron spectroscopy in an UHV apparatus that allows oxidation of freshly deposited amorphous carbon films. The dissociative sticking coefficient of oxygen is ~10-6 and the oxidation kinetics can be described by a Langmuir-Hinshelwood mechanism. The vapor phase lubrication of amorphous carbon film has been emulated by absorbing (CF3CF2)2O and CF3CH2OH that serve as models for the fluorinated ether backbone and hydroxyl endgroup of PFPE lubricants. No decomposition was observed. The desorption energy of the hydroxyl endgroup is sensitive to the oxidation of the amorphous carbon film by air or O2. The increase of the desorption energy is proportional to the oxygen content on the amorphous carbon film surface. This indicates that the interaction between PFPE lubricant and the amorphous carbon film can be tailored by controlled oxidation of the amorphous carbon film. Vapor phase lubrication not only helps to probe the bonding mechanism of PFPE lubricant with amorphous carbon film, but also proposes a lubrication method which allows people to design and tune the bonding of PFPE lubricants with amorphous carbon films by controlled oxidization.

2:40pm **AS+TF-ThA3 Laser Processing of Polymer Nanocomposite Thin Films**, ***A.T. Sellinger,*** *E.M. Leveugle, G. Peman, L.V. Zhigilei, J.M. Fitz-Gerald,* University of Virginia

Current biotechnology and sensor research has enhanced the drive to establish viable methods for depositing high quality organic and polymer thin films. In this research, solid targets of polystyrene (PS), poly(methyl methacrylate) (PMMA), and poly-lactic co-glycolic acid (PLGA) were prepared for room temperature pulsed laser deposition (PLD). Likewise, frozen targets consisting of varying concentrations of PS, PMMA, and PLGA dissolved in a volatile matrix material were prepared for matrix assisted pulsed laser evaporation (MAPLE). Carbon nanotubes (CNTs) were subsequently added to both PLD and MAPLE target systems in upwards of 20 wt.% loading for deposition of polymer nanocomposite films. Targets were ablated using a 248 nm KrF laser at fluences ranging from 0.025 J/cm@super 2@ to 1.0 J/cm@super2@. In addition, polymer concentration in MAPLE targets was varied between 1 wt.% and 10 wt.% relative to the matrix solvent. Films were deposited on Si, SiO@sub 2@, and NaCl substrates at room temperature in an Ar atmosphere. The chemical properties of the films deposited by both methods varied significantly from the native, with changes in molecular weight on the order of 75%. While PLD produced films generally showed little observable morphology, MAPLE deposited films typically showed evidence of blistering. A molecular dynamics (MD) program developed to simulate ablation of a frozen target during MAPLE was utilized to model and predict experimental phenomena. Particularly, the ejection of clusters of PMMA and volatile matrix molecules was studied and related to the observed morphology of the deposited films.

3:00pm **AS+TF-ThA4 Thin Films of Fe on Pt(111) : Alloy Formation**, ***M. Yoshimura***, *S. Komaru, K. Ueda,* Toyota Technological Institute, Japan

Addition of transitional metals to Pt catalysts has been known to improve catalytic efficiency. Pt-Fe bimetallic catalysts attract attention in industrially important fields such as polymer electrolyte fuel cell (PEFC).@footnote 1@ Modification of geometrical and electronic structure of Pt surface would be responsible for the improved efficiency. Recently, Jerdev et al. examined Fe-Pt alloying processes by XPS and LEED.@footnote 2@ They found that heating thick Fe films on Pt result in an ordered alloy layer of 2 x 2 structure. However, detailed atomistic study has not been carried out. Here, we report scanning tunneling microscopy (STM) study on the growth of Fe on Pt(111) and the alloying processes by heating. All experiments were performed in ultrahigh vacuum of a base pressure below 2 x 10@super -8@ Pa. Pt surface was cleaned by cycles of Ar@super +@ ion bombardment at 1 keV and subsequent annealing in vacuum to 1300 K. Surface cleanliness was monitored by XPS and STM observation. Fe (99.999 %) was deposited onto the Pt(111) surface by an e-beam evaporator. STM observation was carried out at room temperature, and electrochemically etched tungsten tips were used. After 1.1 ML Fe deposition at room temperature, two-dimensional Fe islands up to three stories were formed and the Pt substrate was still visible, which is reasonable in terms of surface free energy. After annealing to 800 K, diffusion of Fe occurs and a network structure consisting of Fe and Pt atoms appears on the surface. After annealing to 1070 K, 2 x 2 structure was locally formed at the step edges, which corresponds to the reported alloy structure by Jerdev et al. It is also found that the surface includes defective structure as well as phase boundaries. The growth of Fe on Pt(111) as well as detailed alloying processes are discussed. @FootnoteText@ @footnote 1@ L-J. Wan et al., Chem. Comm. 2002 (2002) 58.@footnote 2@ D. I. Jerdev et al., Surf. Sci. 513 (2002) L391.

3:40pm **AS+TF-ThA6 Scanning Auger Microscopy of Alkylated Crystalline Silicon(111) Surfaces**, ***H.M. Meyer III,*** Oak Ridge National Laboratory; *L.J. Webb,* California Institute of Technology; *D.F. Paul,* Physical Electronics; *N.S. Lewis,* California Institute of Technology

Silicon materials used in solar energy conversion devices must be prepared to a high degree of purity in order to prevent recombination of the photoexcited electron-hole pair. Recently, a wet chemical alkylation technique has been shown to result in an atomically-flat surface covered with bound alkyl groups preventing complete formation of silicon oxides at the surface even after exposure to oxidizing environments over long time periods. Surface charge carrier recombination velocities remain remarkably low, indicating that silicon oxides forming do not result in charge carrier trap states as previously observed on un-passivated, hydrogen-terminated Si(111). These results indicate that any detectible oxide is not growing in a uniform manner, but rather in small patches leaving large portions of the alkylated surface free of any oxide. Extensive x-ray photoelectron spectroscopic evidence on the chemical structure and reactions of these passivated surfaces has been collected over large areas of the surface. Scanning Auger microanalysis was used to map the oxidation of alkylated Si surfaces to determine if it was proceeding uniformly or in discrete areas. Preliminary results using a PHI 680 Scanning Auger Nanoprobe showed distinct patches of oxide less than 100 nm in width. The orientation of the oxide patches suggested that growth was occurring along step edges between flat terraces, leaving the terraces themselves chemically unaltered. Comparison of these early results were made with data obtained using PHI 700 Field Emission Scanning Auger Nanoprobe, a higher performance Auger Electron Spectroscopy (AES) system. The PHI 700 Schottky field emission optics offer Auger spatial resolution of less than

8nm and made it possible to image the oxide features more clearly. Further, the PHI 700 was able to image areas with comparable resolution as the PHI 680, but with less current and therefore less alteration of the oxide due to electron beam reduction.

4:00pm **AS+TF-ThA7 Electron Beam Induced Processes for Repairing Defects on Quartz Masks*, M. Fischer*,** *J. Gottsbachner, S. Mueller, E. Bertagnolli, H.D. Wanzenboeck,* Vienna University of Technology, Austria

For conventional optical masks focused ion beam (FIB) has become a popular tool for repairing defects. A fundamental problem of using FIB for mask repair is the implantation of Ga-ions and the damage of the substrate surface. Especially for 248 nm and 193 nm lithography electron beam repair avoids the transmission loss which is generated by the implanted Ga-ions. This paper presents some promising results that demonstrate the feasibility of using electron beam induced processes for repairing defects on quartz masks. A commercial scanning electron microscope with a tuneable acceleration voltage of 0,1- 30 kV equipped with a gas inlet system was used to investigate a siloxane based deposition process of silicon oxides on quartz glass substrates. Siloxane together with an oxidizing reagent is decomposed by the electron beam on the surface of the quartz glass substrate. The process was optimized towards high material purity. A chemical investigation of the deposited structures was performed by Auger electron spectroscopy and EDX. The optical properties of the deposited material were investigated by infrared, UV/Vis and AIMS measurements. The influences of process parameters such as precursor gas combinations, precursor gas composition ratios, and electron beam parameters on the optical transparency were discussed. The electrical features of the deposited silicon oxide as dielectric material were tested with a metal-insulator-metal setup. A correlation between the electrical properties, the material purity and the optical transparency of the deposited silicon oxide structures is discussed. This work is considered a solid bias for a better understanding of electron induced deposition of silicon oxide and path the way of a further process optimization of this quartz glass mask repair technique.

4:20pm **AS+TF-ThA8 Extending Defect Root-Cause Analysis to sub-100nm in-film Particle Contamination*, C. Lazik*,** *Y. Uritsky,* Applied Materials, Inc.

Scanning Electron Microscopy (SEM) in combination with Energy Dispersive X-Ray Spectroscopy (EDX) is essential for effective root-cause particle analysis in the semiconductor industry and forms the core of commercial defect review tools (DRT). In many cases, however, particularly for small (<0.1µm) in-film particles located within or under deposited films, the standard DRT approach is less straightforward because of (i) uncertainty in the position of the defect 'core' within the film and/or (ii) poor SEM contrast in dielectric films. These effects ultimately limit the assurance that the electron beam is focused on the defect "core" during automated spectrum acquisition. In addition, the defect geometry may preclude the subtraction of an EDX background spectrum. We present here several examples related to the analysis of ~ 50nm "bump" defects, each of similar surface morphology, encountered following the deposition of one or more dielectric films. Providing a suitable analysis of the particle cross-section enabled the classification of the defect based upon its location within the film-stack as well as the defect formation mechanism (e.g. gas-phase nucleation, arcing). In each case, the defects were analyzed initially by top-down SEM/EDX followed by cross-sectional analysis using the focused ion beam (FIB) with only limited success. Extended Auger (PHI Smart-300 DRT) and/or TEM analysis on FIB prepared samples proved critical in yielding accurate root-cause analysis. Details will be discussed in presentation, but the general conclusion is that the integration of FIB/TEM into the modern DRT platform is necessary.

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