

Tuesday Afternoon Poster Sessions, November 16, 2004

Advanced Surface Engineering Room Exhibit Hall B - Session SE-TuP

Poster Session

SE-TuP1 Ab-Initio Structural Properties and Stress-Deformation Analysis by Rheological Modeling of Fracture of Diamonds-Containing Nanocomposites, *M.V. Kireitseu*, University of New Orleans; *I.O. Nedavniy*, Institute of Strength Physics and Materials Science, Siberian Branch of Russian Academy of Sciences, Russia

The principal goal of the paper is to study fundamentally pure interfaces diamond-like C / metal or polymer structure (matrix). Diamonds nanoclusters with effective sizes between 2 nm and 50 nm were used. The present work describes study of stable atomic structure of diamond-like nanoclusters embedded into metal or polymer matrix. Diamond nanoparticles have spherical, fullerene or pyramidal-type shapes and diamonds fibers as well. Local structure deformation (rearrangement) of diamonds/metal interfaces was principal object of researches. Energy or band structure and density of electronic states analysis were investigated. Possible defect or surface states were described. Ab initio LCAO (HF or DFT) schemes, LASTO (Linear augmented Slater-type orbital method), MLASTO (Modified linear Slater-type orbital method), ab initio DFT LDA (FLAPW, LASTO, pseudopotential) super cell schemes and semi empirical LCAO (AM1, PM3, PM5, etc.) methods were used for diamonds nanoclusters with effective size less than 2 nm. It was found that embedded diamonds might be reconstructed in a fullerene-like manner. Fundamental parameters for tight-binding and molecular dynamics calculation schemes were further based on computer simulation at each direction can be easily developed for better understanding and faster calculations. Study of size dependent mechanical and structural properties revealed at what scale principal fundamentals may give reliable explanations at examining fracture mechanics of nanostructured composites. We also present was to present the modified linear augmented Slater-type orbital method (LASTO) for solving Schrodinger's equation in nanodiamonds crystals with arbitrary atoms per unit cell. It follows to expect this method will allow more efficiently calculating an electronic band structure and structural energy difference for vary transition nanocomposites. As a result overlap matrix and hamiltonian matrix elements were derived in details.

SE-TuP2 Field Emission Enhancements from C-implanted Molybdenum, *N.D. Theodore*, *R.A. Outlaw*, *B.C. Holloway*, *D.M. Manos*, College of William and Mary

The vacuum microelectronic industry is developing large, compact field emitter arrays in high-brightness devices for flat panel displays and microwave tubes. Refractory carbides are materials of interest due to their reduced work function, stable emission, high current capability, and robustness. The purpose of this study was to better understand field emission enhancements observed in molybdenum carbide by comparing it to pure molybdenum. According to Fowler-Nordheim (FN) theory, field emission is determined by two parameters, the work function of a material and its surface roughness. Slope-intercept plots of the resulting FN data are then used to separate the effects of these variables. In this experiment, we deposited molybdenum onto both flat silicon samples and triangular silicon gratings that possessed a tip radius of approximately 10 nm. We then created molybdenum carbide by implanting carbon using plasma immersion ion implantation. Carbide formation was verified using XPS; both the C1s peak and the Mo3d peaks were shifted. Depth AES analysis determined that the carbon-implant extended ~45nm from the surface. The field emission results quantify the observed differences in work function and in emission current stability, and correlate these to variations in surface layer compositions and geometry. Moreover, flat samples did not exhibit repeatable electron emission. We conclude that in flat samples, field emission from surface defects dominates the measured emission current, and this mechanism is not explained by Fowler-Nordheim theory.

SE-TuP3 Chemisorption to the Oxide Surface of Aluminium Metal: The Competing Roles of Hydroxide Formation and Adventitious Contamination, *M.R. Alexander*, The University of Nottingham, UK; *G.E. Thompson*, UMIST, UK

An understanding of the interaction of organic functionalities with the surface of aluminium is required for development of a range of application areas including environmentally friendly adhesion promoters, superhydrophobic architecture, MEMS and NEMS. Formation of numerous types of organic monolayer on aluminium has been reported in the

literature, the first of which predates that of alkane thiols on gold.@footnote 1@,@footnote 2@,@footnote 3@ Whilst the instability of the aluminium oxide surface in ambient conditions is widely recognised, quantification of the changes in surface hydroxyl group concentration and carbonaceous contaminant from the ambient atmosphere have only recently been achieved using X-ray photoelectron spectroscopy (XPS).@footnote 4@,@footnote 5@ Previous work has identified the significant influence of an ambient laboratory atmosphere on assembly of alkyl carboxylic and phosphonic acid monolayer.@footnote 6@,@footnote 7@ Here, we investigate the influence on chemisorption from dilute solutions of such molecules using controlled atmospheric exposure prior to assembly. The order of the resultant SAMs is used as a relative measure of the reactivity of the surface to the headgroups. Water contact angle, IRRAS and XPS are used to provide information on the chemistry of the oxide surface and the order the monolayer assembled onto this surface. Differences are rationalised using the competitive processes of hydroxyl adsorption site formation and adventitious contamination blocking of such sites developed in the earlier work. @FootnoteText@ @footnote 1@ Bigelow, W et al. Colloid Sci 1946; 1: 513.@footnote 2@ Timmons, C, Zisman, W. JPhysChem 1965; 69: 984.@footnote 3@ Allara, DL, Nuzzo, RG. Langmuir 1985; 1: 45.@footnote 4@ Alexander et al. SIA 2000; 29: 468.@footnote 5@ Alexander et al. SIA 2003; 35: 649.@footnote 6@ Alexander, MR et al. ASST:ATB Metallurgie, Brussels, 2004; 60.@footnote 7@ Pertays, K et al. SIA In Press.

SE-TuP4 Microstructure and Properties Changes in CrN Films Influenced by an Implanted Cr Interlayer, *H.-Y. Chen*, National Tsing Hua University, Taiwan; *S. Han*, National Taichung Institute of Technology, Taiwan; *H.C. Shih*, National Tsing Hua University, Taiwan

A chromium interlayer was implanted in (100) silicon using a metal vapor vacuum arc ion implanter, and then CrN films were deposited by cathodic arc plasma deposition. Two types of the CrN films denoted as CrN/Cr/Si (with an implanted Cr interlayer) and CrN/Si (without an implanted Cr interlayer). XRD patterns revealed the presence of CrN (220) preferred orientation for both cases, indicating that regardless of whether the films included a Cr interlayer, the preferred orientation and the columnar structure of CrN films were unchanged. But the interlayer induced finer grain structure and reduced the residual stress of the films from -8.1 GPa to -2.8 GPa, which relaxed by up to 65%. Moreover, the CrN films with a Cr interlayer were less hard and exhibited a higher electrical resistivity than those without. Both specimens were annealed at 500°C for 2 h in an N@sub 2@/H@sub 2@ atmosphere to elucidate the thermal stability of the CrN film. The CrN/Si revealed a phase transformation from CrN to Cr@sub 2@N during annealing, which is due to largely stress relaxation in the film. A implanted Cr interlayer can effectively relax the residual stress in CrN films and prevent a occurrence of phase transformation during annealing. The correlation between the properties and microstructure of the CrN film is also discussed.

SE-TuP5 Oxidation Behavior of Titanium Nitride Films, *H.-Y. Chen*, *H.C. Shih*, National Tsing Hua University, Taiwan

TiN films have been a universal coatings due to their high melting points, extreme hardness, high chemical stability, golden color and so on, and the oxidation of films occurs in the hostile environmental. The oxidation mechanism of TiN films has investigated by their crystal structure and morphology using XRD and Raman scattering spectroscopy and FESEM. TiN films were synthesized by using cathodic arc plasma deposition technique because its high ionization ratio, fast deposition rate. After films deposition, the films were annealed in static air at 500°C to 800°C for 2 h. The XRD and Raman spectra indicated that the rutile TiO@sub 2@ was identified above 600°C and its relative intensity rapidly increased with temperatures. The films completely oxidized into rutile phase above 700°C. The as-deposited TiN films were columnar structure. Nevertheless, the oxide layer appeared on the top of columnar TiN films above 600°C. Furthermore, the microstructure of the oxide was porous with the pore size of several nanometers within the oxide layer at 600°C. As increasing annealing temperature, the oxide morphology possessed elongated grain structure with the aspect ratio of ten and the pore size within the oxide layer ranged in several ten nm, which indicated the densification occurring. Form the results, the oxide layer obvious grow inward with temperatures, which indicates the oxidation of TiN films is inward oxidation, meanwhile the oxide thickness was measured with temperature and the active energy for the oxidation was deduced, which was 110±10 kJ/mol.

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SE-TuP6 Effect of Ion Irradiation during Deposition on the Structure of Alumina Thin Films Grown by Plasma Assisted Chemical Vapour Deposition, D. Kurapov, O. Kyrylov, J.M. Schneider, RWTH Aachen, Germany

Alumina thin films deposited by plasma assisted vapour deposition were studied with respect to the structure and composition by X-ray diffraction and electron probe microanalysis, respectively. Alumina thin films were deposited on hot work tool steel AISI H11 at a growth temperature of 500 to 600 °C. The ion energy was affected by controlling the substrate power density from 2.7 to 6.6 W/cm², which corresponds to the bias potential range from 720 to 905 V. Within the investigated process window the following characteristic phases could be identified : amorphous alumina, γ -alumina, α -alumina as well as mixtures thereof. The alumina phase formation was found to be strongly influenced by deposition temperature and power density at the substrate. The influence of the power density is discussed with respect to the ion energy distribution, which is estimated based on the charge exchange model of Davis and Vanderslice.

SE-TuP7 Microstructural Evolution on the Oxidation of Arc Ion-plated TiN/(Ti,Al)N Superlattice Coatings, M.H. Shiao, National Science Council, Taiwan; C.C. Wang, F.S. Shieu, National Chung Hsing University, Taiwan

TiN/(Ti,Al)N superlattice coatings with a TiN interlayer were prepared by a dual-cathode arc ion plating system on AISI 304 stainless steel. Two targets of Ti and Ti₅₀Al₅₀ were used for the vertically opposed cathodes. Oxidation of the nitride-coated steel was carried out at the temperature of 800°C in air for 60 min. The microstructure and chemistry of the as-deposited and oxidized specimens was characterized by X-ray diffraction, scanning electron microscopy (SEM), transmission electron microscopy (TEM), and Auger electron spectroscopy (AES). XRD result shows that nitride coatings have strong (111) preferred orientation, and TiO₂ phase appeared in the oxidized specimen. Cross-sectional TEM reveals a TiN/(Ti,Al)N superlattice structure with a period of 7.7 nm examined by the selected area diffraction (SAD). In the oxidized specimen, there are dense amorphous Al₂O₃ layer and underlying porous TiO₂ layer on the surface of the coating. Auger depth profiling of the oxidized specimens reveals that the Al element concentration near the surface layer is much higher than that in the nitride coating, and further confirmed the cross-sectional TEM results.

SE-TuP8 Development of Amorphous Layered Structures by Laser Irradiation for Enhanced Corrosion Resistance, J.G. Hoekstra, P.M. Mackey, N. Ünlü, G.J. Shiflet, J.R. Scully, J.M. Fitz-Gerald, University of Virginia

Al and Fe based amorphous materials attract attention for their desirable mechanical hardness and corrosion resistance. This study investigated the atomic scale relationships of amorphous layer formation as a function of chemistry and pulsed laser irradiation conditions in Al-Co-Ce and Fe-Mo-Cr-Mo-C-B alloys. The primary objective was to laser surface modify a continuous, homogenous, amorphous layer on a crystalline substrate. Irradiation of a material with a short laser pulse, 3-100 ns, establishes rapid melting and solidification velocities at the surface, 10⁷-10⁸ K/s and 10⁻¹-10¹ m/s respectively. A KrF excimer laser ($\lambda = 248$ nm, 25 ns at FWHM, 15 Hz) operating with fluences ranging from 0-10 J/cm² irradiated a target surface with corresponding velocity between 0-50 mm/s in a controlled atmosphere ranging from 50-500 mTorr. Characterization was performed by SEM, EDS, XRD, and electrochemical analysis. The resulting microstructures were correlated with electrochemical analysis and devitrification behavior.

SE-TuP9 Nanomechanical Testing Using Noncontact Acoustic Technique, O.G. Lysenko, N.V. Novikov, V. Grushko, Institute for Superhard Materials, Ukraine

The new techniques for noncontact measurements of acoustic waves and vibration parameters, such as atomic force acoustic microscopy and tunneling accelerometer have been developed the last years. We present the experimental technique that uses noncontact acoustic method to nanoscale surface characterization. A pulse laser is used to generate surface acoustic waves in a sample. Two SPM tips detect phase and amplitude of the acoustic waves. This approach makes it possible to measure acoustic transit time within small areas of a samples. From the velocity of the surface waves the elastic modulus can be calculated. Also it is possible to identify the onset of plastic flow in the thin films. A theoretical model embracing the most significant features of wave phenomena at nanoscale level is described. Laboratory experiments have

displayed results that are in agreement with those obtained from theoretical model.

SE-TuP10 Characterizations of the Surfaces of Shocked-Bi-Pb-Sr-Ca-Cu-O Superconductor Particles for a Magnetic Sensor, H.K. Kezuka, Tokyo University of Technology, Japan; K.Y. Yamagata, M.I. Itoh, Kinki University, Japan; T.S. Suzuki, Tokai University, Japan; M.K. Kikuchi, Tohoku Fukushi University, Japan; T.A. Atou, M.K. Kawasaki, K.F. Fukuoka, Tohoku University, Japan

Recently, there has been an increased need for a highly sensitive magnetic sensor. Bi-Pb-Sr-Ca-Cu-O (BPSCCO) oxide superconductors are very attractive material as a highly sensitive magnetic sensor in which magnetic sensitivity of the sensor is found to be about 27 %/(10⁴ T) using BPSCCO powders pasted for constructing the magnetic sensor. Characteristics of BPSCCO superconductor depend sensitivity on the grain growth temperature. Researches for applications of high-T_c BPSCCO superconductor particles have been made to overcome the weak-link problem around the grain boundaries for higher J_c. Shock compaction for BPSCCO superconductor particles have been investigated by shock compaction technique. After the shock compaction characterizations of as-shocked BPSCCO superconductor particles are investigated by X-ray diffraction (XRD)-analysis for the measurements of lattice parameters and SQUID-measurements for the susceptibility. The grain growth with growth steps of as-shocked BPSCCO superconductor particles are observed by AFM (Atomic Force Microscope) and SEM (Scanning Electron microscopy). The particle size distribution was measured by a laser diffraction size analyzer, which employed laser diffraction and scattering methods. In this measurement, use was made of an isopropyl alcohol for dispersing the BPSCCO particles. The average particles is found to be about 4.2 μ m. The specimens were pressed into pellets with 10 mm in diameter and around 1 mm in thickness. These pellets were tightly encased in stainless containers. Shock compaction experiments were carried out under the shock pressure of around 2-5 GPa by using a propellant gun-system. From XRD-analysis, it is found that Bi-2223 phase is appeared clearly after the shock compaction process under 2-5 GPa with Bi-2212 phase. The Amplitude Mode image shows the surface details better than the unfiltered Height Mode images for BPSCCO particles. Tapping AFM image of the surface and edges for annealed BPSCCO particles at 850 °C for 96 hr after shock compaction. It is found from the section analysis that the vertical distance around edge area varies from 86 to 478 nm. BPSCCO superconductors, the Bi-2223 structures of as-shocked BPSCCO probably have shock-induced defects by the shock compaction. The ΔM (the difference of field cooling and zero field cooling at 20 Oe) which is proportional to J_c, is measured by SQUID magnetometer. As a result, from the measurements of the susceptibility by SQUID magnetometer, it is found from the susceptibility measurements as a function of temperatures that ΔM clearly increases to 0.073 (emu/g) for as-shocked BPSCCO after annealed at 850°C for 48 hr, which seems to indicate the new pinning centers around the grain boundaries caused probably by the new defects of vacancies. The shocked specimens was compacted in platelet disk with a high crystal density over 97 %. The as-shocked specimen showed an oriented crystal structure with large grains of 3-5 μ m in length partly for shocked specimen by 2-5 GPa. The ΔM (0.073 emu/g) of shocked specimen after annealing at 850° C for 48 hr became higher than that of starting specimen. These increase of ΔM indicate the introduction of a new pinning center through the shock compaction and annealing processes. The Amplitude Mode image of AFM show large edge effects which reflect the amplitude changes. Also from the section analysis of AFM, the vertical distance of 86-478 nm is obtained around the edge area in grains for annealed BPSCCO after shock compaction.

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