Thursday Evening Poster Sessions, November 16, 2006

Advanced Surface Engineering Room 3rd Floor Lobby - Session SE-ThP

Advanced Surface Engineering Poster Session

SE-ThP1 Study of Mechanical Properties of Tungsten Nitride and Tungsten Carbide Hard Coatings, *E.C. Samano, W. De la Cruz, G. Soto,* CCMC-UNAM, Mexico

Transition metal nitride and carbide coatings are a good alternative to be used as protective layers against wear because transition metals are hard materials with a high melting point. Tungsten nitride and carbide coatings are an excellent choice to prevent wearing of cutting tools, structural components, etc. Their mechanical properties can be tailored by controlling the nitrogen and carbon content, in each case, during film processing. The investigation on the relationship between thin film preparation conditions and mechanical properties of tungsten nitride and tungsten carbide is presented in this work. We report the growth of tungsten nitride and carbide coatings grown on stainless steel substrates by reactive dc magnetron sputtering in the ambient of N@sub2@/Ar and CH@sub 4@/Ar gas mixtures, respectively, at various pressures and magnetron system input powers. The composition of the films is determined by XPS and film crystallinity by XRD. The mechanical properties of the films are studied as a function of gas pressure and magnetron input power. The average hardness of the deposited coatings were found to be in the 31 to 37 GPa range for tungsten nitride and in the 30 to 41 GPa range for tungsten carbide.

SE-ThP3 Ti@sub x@Al@sub y@Si@sub z@N Films for Super-Hard Coatings Deposited by Reactive Co-Sputtering using Ti, Al and Si Targets, *A. Miyamura*, *M. Yamaguchi*, Aoyama Gakuin University, Japan; *K. Hattori*, National Institute of Advanced Industrial Science and Technology, Japan; *Y. Sato*, *Y. Shigesato*, Aoyama Gakuin University, Japan

TiNx is one of the promising hardcoating materials for its high toughness, high hardness and high chemical stability. Recent research goes to add the third element in order to make it extremely harder, higher melting point and higher thermal stability. For example, TixAlyN increases hardness by solid solution strengthening, whereas TixSiyN increases hardness by selfconstructing nanocomposite structure. In order to attain more outstanding properties than the both ternary systems, we deposit TixAlySizN super hard films by three cathode co-magnetron sputtering systems. This system can adjust a composition of Ti, Al and Si in the film by controlling the input power ratio of the three cathodes mostly following the additive properties. As a result, TixAlySizN films with various compositions were deposited on silicon or glass substrates. For all the depositions, Ar was used as sputtering gas and N@sub 2@ was introduced as reactive gas. Crystallinity and surface morphology of the films were analysed by X-ray diffraction (XRD) and atomic force microscope (AFM). Compositions were estimated using Xray photoelectron spectroscopy (XPS) and electron probe micro analyzer (EPMA). A nano-indentation equipped with Berkovich diamond indentor using a continuous stiffness method was used to measure the hardness and Young's modulus along the depth of the TixAlySizN films. With increasing Si concentration, intensity of XRD peaks decreased and the films became amorphous over nearly 20% of Si concentration. It was clarified that the hardest condition could be obtained when titanium concentration was 2-3 times of aluminum concentration and silicon concentration was about 10-20 %. For these films the nanocomposite structure was clearly observed by TEM. This work was partially supported by a Grant-in-Aid for the 21st COE Program from the Ministry of Education, Culture, Sports, Science and Technology (MEXT) of the Japanese Government.

SE-ThP6 Pulsed AC Plasma Polymerisation for the Chemical Modification of Glassy Carbon Surfaces in Model Composite Interface Studies, J.M. Drews, The Technical University of Denmark, Denmark; S. Goutianos, P. Kingshott, N. Rozlosnik, Risoe National Laboratory, Denmark; S. Hvilsted, The Technical University of Denmark; K. Almdal, B.F. Soerensen, Risoe National Laboratory, Denmark

Pulsed AC plasma polymerization was used for surface modification of glassy carbon (GC) substrates, which are chosen as a model for the surface chemistry of carbon fibres used in composites. The pulsed AC plasma system, which is custom built, is powered by a 50 Hz 2-phase AC power supply from the grid and phase 1 is shifted 180° from phase 2. The resulting voltage between the electrodes (the plasma voltage) is the sum of phase 1 and 2. Depending on pressure and gas composition, a voltage of around 250 V is needed to ignite the plasma, resulting in a (100 Hz) pulsing plasma

current that has an effective power input of less then 1 W/L. This opens up the possibilities of generating plasma polymer layers by plasma-induced radical polymerisation. We utilise maleic anhydride (ppMAH) and 1,2methylenedioxybenzene (ppMDOB) to generate both homo-plasma polymer and co-plasma polymer layers surfaces, in the power range from 0.3 to 1.3 W/L, to generate stable layers with varying degrees of functional integrity. X-ray Photoelectron Spectroscopy (XPS) and Attenuated Total Reflectance Fourier Transform Infrared spectrometry (ATR-FTIR) were used to monitor surface chemistries, and toluidine blue (TB) staining to determine the number of reactive acid groups after hydrolysis. Atomic Force Microscopy was used to monitor both topography and layer thicknesses using a polymeric masking method. The results show that the properties of the plasma polymer layers can be subtly varied by the plasma conditions. Unexpected results were obtained using the TB staining, and indicate that the hydrolysis of MAH groups is much more difficult to achieve than previously reported. Finally, the interfacial fracture properties were determined using a Double Cantilever Beam (DCB) method. It was found that differences in fracture energy are related to differences in surface properties, and that in all cases, the plasma polymer coatings increased the fracture energy, compared to the uncoated GC.

SE-ThP7 Integrating Aligned Nanorod Array onto Optical Fibers, J.G. Fan, Y.J. Liu, Y.P. Zhao, University of Georgia

We report a modified oblique angle deposition (OAD) method to prepare nanorod arrays onto optical fibers or cylindrical objects. By rotating the optical fiber with a proper tilting angle, aligned nanorods from materials, such as metals, semiconductors and metal oxides, have been successfully deposited onto optical fibers. With a slight modification, multi-layer film/nanorod and nanorod/nanorod structures have also been demonstrated. Surface enhanced Raman spectroscopy of trans-1,2-bis(4pyridyl)ethane (BPE) molecules on Ag nanorod array on a fiber optical tip is demonstrated.

SE-ThP8 Protective Oxide Layers on Austenitic Stainless Steels (316L and D9) in Molten Lead Service: Formation and Reformation, Structure and Dynamics, A.L. Johnson, J.W. Farley, D. Koury, B. Hostermann, J. Welsh, T. Ho, University of Nevada, Las Vegas; L. Ma, Harry Reid Center; U. Younas, University of Nevada, Las Vegas

Liquid lead based alloys are of current interest due to their use as nonmoderating coolants for fast neutron systems for power generation and nuclear waste transmutation. Unfortunately liquid lead corrodes steel by dissolution of the alloy's components. This corrosion may be mitigated by the injection of low levels of oxygen in the lead to form protective oxide layers. However the initially formed oxide layer is subject to failure and must reform when damaged under operational conditions. We have been investigating the structure and composition of the oxide layers formed on steels by oxygen controlled Lead Bismuth Eutectic (LBE) using XPS, SEM/WDS, and TEM. We find that there is an initial crystalline oxide (high Cr, low Fe) that converts to a complex bilayer (magnetite over (Fe,Cr)@sub 3@O@sub 4@) when damaged. Detailed investigation of the morphology and composition of these layers indicates the relative mobility of the alloy components in the near surface region and suggests a novel mechanism for the previously observed improvement in corrosion resistance due to cold working of the surface and near surface region. We will discuss this work in the context of current work with oxide layers formed by oxide generating species (Al, Si, and Y) introduced by sputter deposition or alloying.

SE-ThP9 Two Dimensional Micro Honeycomb and Nanoring Array, D. Jia, C. Shaffer, J. Weyant, A. Goonewardene, Lock Haven University of Pennsylvania

Polystyrene microspheres with 10, 2, 1 and 0.56 micron sizes were used to prepare monolayer as patterning for sputtering deposition. Gold, silver and platinum metal sources were used as sputtering deposition source. Two dimensional micro-honeycomb and nanoring metallic arrays were fabricated on glass and silicon substrate. No ring structures were obtained for platinum and silver deposition. Transition from honeycomb to ring structure was found for deposition gold. For 10 micron size microspheres, honeycomb structures were dominant and no ring structure was found. For 2 micron size microspheres, honeycomb and ring structures were found to co-exit. For size smaller 1 micron, ring structures. Laser diffractions were studied for these structures.

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SE-ThP10 Influence of the Negative Oxygen Ions on the Structure Evolution of Transition Metal Oxide Thin Films, S. Mráz, J.M. Schneider, RWTH Aachen University, Germany

The energy distributions of O@super -@ ions of magnetron sputtered Nb, Ta, Zr, and Hf in an Ar/O@sub 2@ atmosphere were measured as a function of the oxygen partial pressure. Three ion populations were detected in the plasma: low, medium and high energy ions. The ion energy distribution functions were compared to distributions obtained based on sputtering theory. If the surface binding energy is assumed to be equal to the heat of formation, good agreement between the experiment and theory was achieved. From correlating the measured ion energy distributions with previously published phase stability data,@footnote 1@ it can be deduced that large fluxes of medium and high energy O@super -@ ions enable formation of crystalline transition metal oxide thin films during low temperature growth. The here presented data may be of general relevance for understanding the structure evolution of thin oxide films. @FootnoteText@ @footnote 1@ Ngaruiya et al., Appl. Phys. Lett. 85(5), 748 (2004).

SE-ThP11 Influence of the Normalized Ion Flux on the Constitution of Al@sub 2@O@sub 3@ Films Deposited by Plasma Assisted Chemical Vapor Deposition, *D. Kurapov, J. Reiss,* RWTH Aachen, Germany; *D.H. Trinh, L. Hultman,* Linköping University, Sweden; *J.M. Schneider,* RWTH Aachen, Germany

Al@sub 2@O@sub 3@ thin films were deposited onto tempered hot working steel substrates from an AlCl@sub 3@-O@sub 2@-Ar-H@sub 2@ gas mixture by plasma assisted chemical vapor deposition (PACVD). During deposition the normalized ion flux was varied through changes in the precursor content while keeping the cathode voltage and the total pressure constant. As the precursor content in the total gas mixture was increased the deposition rate resulting in an increased normalized ion flux from 50 to 480. The constitution, morphology, impurity incorporation and the elastic properties of the alumina thin films were found to be normalized ion flux dependent. These normalized ion flux induced changes in structure composition and properties may be understood by considering surface and bulk diffusion related mechanisms.

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