Thursday Afternoon, November 16, 2006

Applied Surface Science
Room 2005 - Session AS-ThA

Combined Methods or Multiple Methods

Moderator: I.S. Gilmore, National Physical Laboratory

2:00pm **AS-ThA1 A Fundamental Investigation of Erucamide Migration in Polyolefin Matrices***, **J. Chen**, B. Walther, J. Li, T. Hu,* The Dow Chemical Company

Erucamide is the most widely used slip agent in plastic manufacturing processes. It is well know that erucamide migrates toward the surface leading to a reduction in the coefficient of friction (COF), but not much has been published on the details of the surface migration. Our study has shown that the surface layer of erucamide is a function of many factors including the matrix material and thickness, temperature shelf time and other factors. X-ray photoelectron spectroscopy (XPS), time-of-flight secondary ion mass spectrometry (ToF SIMS), atomic force microscopy (AFM) and other technologies were applied to develop fundamental understanding of the erucamide migration mechanism and the correlation with COF. This paper will discuss our fundamental investigations of surface migration of erucamide through polyolefin resins.

2:20pm **AS-ThA2 Phenomenological Relationships Between Chemistry and Mechanical Properties Derived from ToF-SIMS and Nanoindentation Observations***, **G.L. Fisher,*** Physical Electronics*; C. Szakal, N. Winograd,* The Pennsylvania State University*; J.G. Swadener,* Los Alamos National Laboratory

Remarkable correlations between the chemistry and the mechanical properties of polytetrafluoroethylene (PTFE) have been revealed as a function of ionizing radiation fluence. Specifically, changes in the hardness are explained in terms of the macromolecular evolution during @alpha@ particle (5.5 MeV @super 4@He@super 2+@) irradiation. Chemical changes in the surface region of the polymer, i.e. macromolecular evolution, were evaluated using a ToF-SIMS instrument that was equipped with a 20 keV C@sub 60@@super +@ ion probe. The mechanical properties of hardness and elastic modulus were monitored in the near-surface (~2.5 µm) region of the polymer by nanoindentation. Nanoindentation shows that scissioning of the linear polymer chains by @alpha@ particles is offset by crosslinking at moderate doses and leads to a peak in the elastic modulus at ~10@super 8@ Rad. The elastic modulus falls off with continued irradiation due to an increase in molecular mobility that arises by a reduction in the average molecular weight of the matrix molecules (i.e. fragmentation). The magnitude of the hardness indicates that crosslinking continues to a dose of ~5x10@super 9@ Rad and thereafter declines with continued irradiation due to fragmentation. The ToF-SIMS data also reveal a peak in the level of crosslinking at ~5x10@super 9@ Rad that is followed by extensive fragmentation of the matrix molecules. In contrast to nanoindentation, the method by which ToF-SIMS data is used to evaluate the relative degree of crosslinking in a polymer matrix is immature. Therefore, the molecular fragments that were evaluated, the mechanism(s) by which they arise in the mass spectrum, and the treatment of the data will be discussed. These developments represent a significant step forward in the application of ToF-SIMS to address the properties of soft materials.

2:40pm **AS-ThA3 Multi-technique, Multivariate Analysis Methods for Enhanced Sample Characterization***, **J.E. Fulghum**, K. Artyushkova, S. Pylypenko, J.L. Fenton, K.M. Archuleta,* University of New Mexico*; L. Williams,* University of New Mexcio **INVITED**

Characterization of heterogeneous samples frequently requires multi-technique correlations. The ability to acquire images from the same area on samples using multiple techniques provides opportunities for enhanced sample characterization, including using data from one technique to facilitate or confirm interpretation of data from a second technique. A variety of techniques, including AFM, FTIR, XPS and confocal microscopy (CM) have comparable fields-of-view, although spatial resolution and information content differ dramatically. This talk will incorporate a variety of examples of multi-technique correlations including visualization of 3-D polymer chemistry through correlation of XPS, CM and AFM data; fusion of high spatial resolution AFM images with high energy resolution XPS images for enhanced spatial distribution information; quantification of CM image data sets through fusion with XPS quantitative images, and correlation of AFM images with contact angle data. Correlating the data from multiple techniques, as in the examples listed above, requires matching and marking of the sample analysis areas, image registration, multivariate image analysis, image quantification and image fusion. We are currently developing a Matlab-based Graphical User Interface (GUI), that includes all of these steps. The goals of the GUI include managing images from multiple modalities, performing multiple imaging processing steps such as classification and PCA, segmentation, image registration, image fusion, volume reconstruction; providing tools that support flexibility by incorporating new and existing image analysis routines; and providing a simple, yet powerful user interface. The current status and availability of the GUI will be described.

3:20pm **AS-ThA5 Multi-Technique Characterization of Niobium Surfaces for Superconducting Radio Frequency (SRF) Accelerators***, **H. Tian,*** College of William & Mary*; C.E. Reece,* Thomas Jefferson National Accelerator Facility*; M.J. Kelley,* College of William & Mary, US*; S. Wang, L. Plucinski, K.E. Smith,* Boston University*; M.M. Nowell,* Edax Tsl

The accelerator structure for the proposed International Linear Collider (ILC) comprises more than 20,000 Nb cavities operating at 2 K, and having a total internal surface area in excess of 16,000 m2. Upgrade of Jefferson Lab's CEBAF accelerator from 6 GeV to 12 GeV is about to begin. It will require 80 new cavities and will be preceded by refurbishment of about 40 of the original 338. Other applications of RF superconductivity to particle accelerators are being actively explored. The shallow microwave penetration (with local electric fields exceeding 55 MV/m) causes superconducting RF accelerator performance to be strongly influenced by the chemistry, topography and structure of the top several nanometers of the internal surface.@footnote 1@ These are substantially determined by post-fabrication etching and conditioning. We examined single and polycrystal Nb material , etched by chemical polishing and electropolishing and post-treated by extended low-temperature baking at ultra high vacuum condition which are commonly used for cavity production. The combined use of XPS, synchrotron-based (variable photon energy) photoemission, atomic force microscopy (AFM), stylus profilometry and electron backscatter diffraction (EBSD) provides key insights into the effect of post-fabrication treatments on the Nb surface. The improved knowledge of materials aspects of RF superconductivity provides a stronger foundation for future major accelerator projects. @FootnoteText@ @footnote 1@ H. Padamsee, J. Knobloch, T. Hays; RF Superconductivity for Accelerators, Wiley, New York, (1998).

3:40pm **AS-ThA6 Ion Sputtering and the Static Limit for Nanoparticles***, **D.J. Gaspar**, Z. Zhu, A.S. Lea, D.R. Baer, M.H. Engelhard,* PNNL

The behavior of nanomaterials varies in many cases from that observed for bulk materials. During the course of studies on several types of particles and nanostructured materials, we have observed evidence that the extent of damage and material removal rates due to ion sputtering may be significantly different than for continuous films or bulk forms of similar materials.@footnote 1,2@ This presentation will review our efforts to quantify the sputter and damage rates for some particle and nanomaterial systems, including salt particles, iron oxide, titania and ceria nanoparticles and nanoporous silica films. Additionally, we have attempted to correlate measurement of SIMS damage cross-sections in measurements of titania nanoparticles with other measurements of particle structure and chemistry, including Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and X-ray Photoelectron Spectroscopy (XPS). For the data presented here, material removal rates are monitored directly by profilometry, and indirectly by TOF-SIMS, XPS and Auger Electron Spectroscopy (AES). For large aggregates of nanoparticles, TOF-SIMS spectra appear the same as bulk materials. We have extended these measurements to TiO@sub 2@ nanoparticles dispersed by chemical attachment to self-assembled monolayers. We suggest the contributions of geometric factors and energy transfer modified by nanoscale features contribute to observed variations in sputter rates. We examine the relationship of our observations to theoretical efforts including the theory of Bradley and Harper@footnote 3@ and Monte Carlo simulations of nanoscale effects on sputtering.@footnote 4@ @FootnoteText@ @footnote 1@D.J. Gaspar, et al., Surf. Interface Anal., 37 (2005) 417-423.@footnote 2@Baer, D.R., et al., J. Surf. Anal., 12 (2005) In press.@footnote 3@Bradley, R.M. and J.E. Harper, J. Vac. Sci. Technol. A 6 (1988) 2390.@footnote 4@Jurac, S., et al., Astrophys. J., 503 (1998) 247-252.

4:00pm **AS-ThA7 Optimised XPS Depth Profiling of Aluminium Surfaces***, **C. Blomfield**, A.J. Roberts,* Kratos Analytical Ltd, UK*; J.C. Walmsley,* Sintef, Trondeim Norway

Characterization is a key activity within aluminum surface science technology. Corrosion and protection mechanism studies are one of a number of areas of study that also include coatings, joining processes, chemical processing, lubrication, rolling and extrusion. The behavior of trace and impurity elements in aluminum alloys is of interest and it is essential to have effective surface characterization tools to study these. Surface and near-surface segregation studies of model aluminum alloys will be used to illustrate the complementary information that can be obtained from different techniques and how this can be related to the properties of the surface. A combination of low-energy ion gun technology, small area analysis, sample rotation and increased instrumental sensitivity have come together to make XPS particularly suited to analysis of the role of trace elements in surface segregation.

4:20pm **AS-ThA8 Characterization and Metrology Challenges in SiON Gate Thin Films for ULSI Technology***, **G. Conti**, C. Lazik, Y. Uritsky, T.C. Chua, C. Czarnik,* Applied Materials*; S.R. Bryan,* Physical Electronics*; T. Gustafsson, E. Garfunkel,* Rutgers University

Silicon oxynitrides (SiON) films are presently used as a replacement of SiO2 gate dielectric film in oxide semiconductor field effect transistor MOSFET. SiON films can be grown by a variety of processes such as: thermal oxy-nitridation, low energy ion implantation, chemical vapor deposition, and atomic layer deposition. Depending on the growth process and the process parameters, nitrogen can pile up at or near the Si/SiO2 interface, be uniformly distributed throughout the film, or be enriched at the interface of the poly-Si gate electrode. The N distribution significantly affects the electrical properties of the gate dielectric layer; therefore, during process development the physical properties of the SiON film (thickness, composition, and atomic depth distributions) must be determined. Whereas most of the analytical techniques become less effective as film thickness drops towards the 10A range, angle resolved x-ray photoelectron spectroscopy (AR-XPS) becomes a very effective technique in this range. AR-XPS is capable of providing precise thickness and detailed information on elemental and chemical composition. However, extracting a depth distribution from the data requires calculating the angular response for trial models and comparing to the data. Since a fit is not a guarantee of trustworthy, AR-XPS depth profiles results were compared to other surface techniques. We characterized "golden standard" samples for N dose and N profile by TOF-SIMS, and by MEIS, and compared the results to AR-XPS. TOF-SIMS instead predicted a broad and flat profile even for box-like profiles created by CVD and it does not capture any distinct SiO2 layer beneath the SiN layer. MEIS agrees with AR-XPS fit confirming our ability to measure depth-profile non destructively. In addition, preliminary results from TEM/EELS analysis agree with AR-XPS and MEIS results.

4:40pm **AS-ThA9 Ultimate Nanoprobing in UHV: Four independent Scanning Tunneling Microscopes Navigated by High Resolution UHV SEM***, M. Maier, J. Westermann, **T. Berghaus**,* Omicron NanoTechnology GmbH, Germany

A major challenge in Nanotechnology is the incorporation of single nano-devices into larger integrated circuits. Traditional instrumentation suffers from one fundamental issue: How to cover the dimensional range of a fully integrated circuit down to the nanometer range (or even atomic scale) of single devices and have an adequate integrated navigation system. To meet these requirements, we have established and being advancing a new approach to integrating state-of-the-art SPM technology via high resolution electron microscopy and spectroscopy: (1) Bridging dimensions and rapid navigation; (2) Combining different surface analysis methods at the very same sample area to gain complementary information; (3) Integrated position-readout of sample and probe positioning; (4) Pushing each single technology to its inherent limits. The system facilitates four independent Scanning Tunnelling Microscopes and the UHV version of the Zeiss Gemini SEM column with ultimate resolution below 4nm for probe navigation and rapid localisation of sample features or devices. STM imaging is used to pro-actively position and contact the probe(s) on nano-devices. Using STM probe approach technology, a controlled electrical contact is ensured to finally perform a four-point measurement on the nano-scale. Beyond navigation, SEM enables different electron spectroscopy methods to gain magnetic or chemical information on the sample area. Using SEMPA (SEM with polarization analysis) magnetic domain imaging with a resolution in the 10nm range is achieved to allow for correlation of transport properties and domain distribution. Using SAM (Scanning Auger Microscopy) chemical mapping can be achieved with 10nm resolution to correlate transport properties and material composition. Various application examples will be shown to illustrate the system capabilities.

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