Applied Surface Science
Room: Cochiti - Session AS-WeM

New Ion Beam Technologies for Imaging, Sample Preparation and Analysis

Moderator: J.A. Gardella, Jr., SUNY at Buffalo

8:00am **AS-WeM1 Ion Photon Emission Microscopy: A Novel Method for Studying Radiation Effects**, ***J.V. Branson***, *K. Hattar, G. Vizkelethy, C.J. Powell,* Sandia National Laboratories, *P. Rossi,* University of Padua and INFN, Italy, *B.L. Doyle,* Sandia National Laboratories

The development of a new radiation effects microscopy (REM) technique is crucial as emerging semiconductor technologies demonstrate smaller feature sizes and thicker back end of line (BEOL) layers. To penetrate these materials and still deposit sufficient energy into the device to induce single event effects, high energy heavy ions are required. Ion photon emission microscopy (IPEM) is a new technique that utilizes coincident photons, which are emitted from the location of each ion impact to map out regions of radiation sensitivity in integrated circuits and devices, circumventing the obstacle of focusing high-energy heavy ions. The (x,y) coordinates are instead determined with a single photon, position-sensitive detector. Thus, a high energy broad beam can be used to achieve high LETs, while still mapping out radiation-sensitive regions with sufficient resolution. Several versions of the IPEM have been developed and implemented at Sandia National Laboratories (SNL). The initial IPEM was a tabletop system, which utilized a Po-210 alpha source as the incident radiation. The second version has been utilized on the microbeam line of the 6 MV tandem accelerator at SNL, which allows for direct results comparisons between data obtained with a scanned, focused microbeam, and that from IPEM. Another IPEM was designed for ex-vacu use at the 88" cyclotron at Lawrence Berkeley National Laboratory (LBNL). That facility allows for the use of a heavy ion cocktail, with beam energies up to several GeV. Extensive engineering is involved in the development of these IPEM systems, including resolving issues with electronics, event timing, optics, phosphor selection, and mechanics. The various versions of the IPEM and the obstacles, as well as benefits associated with each will be presented. In addition, the current stage of IPEM development as a user instrument will be discussed in the context of recent results.

*Sandia National Laboratories is a multi-program laboratory operated by Sandia Corporation, a wholly-owned subsidiary of Lockheed Martin company, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

8:20am **AS-WeM2 Synthesis and Characterization of Gold Nanocluster-Cavity Pairs in $SrTiO_3$**, *S.V. Shutthanandan, C.M. Wang, B. Arey, W. Jiang, Y. Zhang,* ***T. Thevuthasan***, Pacific Northwest National Laboratory, *G. Duscher,* Oak Ridge National Laboratory

Dispersion of gold nanoclusters in single crystal TiO_2, MgO, and $SrTiO_3$ have been found to influence the optical properties of the materials. One way to make these clusters in these oxide media is through Au ion implantation followed by annealing at high temperatures for extended period of time. The Au clusters generated by this ion beam synthesis method often associated with vacancies as a cluster-vacancy pair in the matrix. These nanometer scale vacancy clusters are generally called quantum antidotes and they are believed to be spatially located at the interface between the Au clusters and the matrix. To verify this proposition, Au nanoclusters dispersed in $SrTiO_3$ single crystals were prepared using ion implantation at 975 K and subsequent annealing at 1275 K for 10 hours. We have used a suite of imaging capabilities including newly developed Helium ion microscopy (HIM) and scanning transmission electron microscopy (STEM) with high-angle-annular-dark-field (HAADF) imaging to understand the structural properties and spatial distribution of the Au and vacancy clusters. The results indicate that gold nanocluster-cavity pairs were formed uniformly throughout the implanted region. Size of the Nanocluster-cavity pairs ranges from 5 to 30 nanometers. In cluster free regions where the Au concentration is low, the HADDF results clearly indicate the substitution of Au for cations. The Au clusters and the cavity show spatial association, indicating a strong interaction during their respective clustering process.

9:00am **AS-WeM4 Material Contrast Mechanisms in FIB and SEM Images**, ***L.A. Giannuzzi***, FEI Company, *M. Utlaut,* Portland State University

The relative contrast of both FIB and SEM images in metals yield a non-monotonic function with target atomic number. Material contrast from FIB images is similar to the material contrast observed from SEM images, with differences that can be directly attributed to particle-solid interaction theory. The non-monotonic particle stopping power and the sputter yield (for those particles that cause sputtering) are directly responsible for material contrast. The term "Z-contrast" is shown to have a different meaning than universally understood.

9:20am **AS-WeM5 A High Brightness Plasma Source for Next Generation FIB, SIMS and Surface Engineering**, ***N. Smith***, *P.P. Tesch, N.P. Martin, R.W. Boswell,* Oregon Physics **INVITED**

Milling speeds with a gallium focused ion beam (FIB) are often much too slow for many sample preparation and surface engineering applications. For example, cross-sectioning stacked-die semiconductor devices, prototyping micro-mechanical structures and delayering IC's for circuit mapping are growing applications that require a milling rate that far exceeds that provided by the gallium FIB.

Furthermore, secondary ion mass spectrometry (SIMS) imaging has been limited to a lateral resolution of 200nm when using an oxygen focused ion beam for high sensitivity surface analysis. Many applications in material science could benefit from an ability to image trace level surface chemistry with <20nm resolution. Example applications include, sub-cellular imaging of trace metals in the brain for neurodegenerative disease studies, analysis of trace element segregation in metal alloys and studying isotope distributions in meteorites and interplanetary dust particles.

In the more general area of direct-write surface engineering, precision milling and deposition with nanometer precision is limited to volumes of $<10^4 um^3$ when using gallium FIB systems. Also, engineered devices must generally be tolerant of high gallium concentrations being implanted in the near-surface region. These are major restrictions when fabricating devices that require nanometer precision across dimensions of several hundred micrometers. Additionally, inherent gallium implantation can render structures bio-incompatible and compromise the electrical properties of many materials.

Here, we review a plasma ion source technology (Hyperion^{TM}) that can provide a focused ion beam capable of milling silicon at a rate of $>5000 um^3/s$ with <4um milling resolution and <20nm imaging resolution with 30keV xenon ions. The same ion source is also readily operated as a high brightness source of oxygen, hydrogen and any inert ions.

By transferring energy to plasma electrons via a RF induction field, it is possible to create a plasma state without a cathodic electrode. This approach can create high plasma densities ($>1x10^{13}$ ion cm^{-3}), with very low mean thermal ion energies (<0.05eV), providing the conditions for an energy normalized beam brightness that now exceeds $1x10^4$ $Am^{-2}sr^{-1}V^{-1}$. This high brightness can be attained with long lifetimes (>>2000 hours), stable beam current (<±0.5% drift per 30 minutes) and an axial energy spread for the extracted beam of 5-6eV and for an array of ion species.

This paper presents FIB and SIMS data from this new ion source. The operating principles of the ion source, the properties of the beam(s) being created and the projected future for this technology are also described.

10:40am **AS-WeM9 XPS Comparison of Ar, Coronene, C_{60}, and Ar Gas Cluster Ion Beam Depth Profiling of Polyimide Films**, ***J.S. Hammond***, Physical Electronics, *T. Miyayama, N. Sanada,* ULVAC-PHI, Japan, *J.F. Moulder,* Physical Electronics, *M. Suzuki,* ULVAC-PHI, Japan, *A. Takuhara,* Kyushu University, Japan

Polyimide thin films have found wide-spread use in many industrial products such as microelectronics and thin film display panels due to their excellent insulating properties, high resistance to heat in manufacturing processes and its excellent flexibility and other mechanical properties. To increase the adhesion of metal films to polyimide substrates, ion beam and plasma surface modification steps are frequently incorporated in the manufacturing processes. It is therefore highly desirable to find a quantitative chemical depth profiling technique to characterize the surface modification layer and the polyimide thin film itself. A comparison of the use of XPS depth profiling of thin polyimide films with Ar, Coronene, C_{60}, and Ar Gas Cluster Ion Beam (GCIB) sputter sources will be presented. The GCIB sputter source produces an $Ar_{2,500}^+$ ion beam with user definable incident beam energy. XPS elemental quantification and chemical state spectroscopy reveals that Ar, Coronene and C_{60}, ion sources produce rapid damage of the polyimide with a wide range of ion gun experimental conditions. Optimized conditions for the incident ion beam energy of the GCIB will be presented to provide minimal chemical state damage during the depth profiling of 100 nm thick films. Results will also be presented showing that the GCIB source can be used to remove Ar^+ induced damage layers.

11:00am **AS-WeM10 XPS Sputter Depth Profiling of Organic Materials Using a Coronene Ion Source**, ***S.J. Hutton***, *C.J. Blomfield, A.J. Roberts, G. Mishra, I.W. Drummond, S.C. Page*, Kratos Analytical Ltd., UK

The X-ray photoelectron spectroscopic (XPS) analysis of thin film or multilayer organic materials has recently been improved by the introduction of cluster ion sources for sputter depth profiling. One such commercially available cluster ion source uses the polyaromatic hydrocarbon (PAH) coronene. This source has successfully produced useful depth profiles of a number of model polymer films [1] [2] and mixed organic systems. [3] Results demonstrate a significant reduction in ion induced damage, as measured by XPS between ion etch sputter cycles, compared with conventional ion sources. High ion yields have been found for several polymers including poly(lactic-co-glycolic acid) (PLGA); Polyacrylic acid (PAA); and polylactic acid (PLA), however, other polymers, such as polystyrene (PSS), appear to be resistant to sputter profiling using cluster ion sources under previously investigated conditions. [4]

In this study we investigate a range of experimental parameters which influence ion yields and surface damage with the aim of optimising sputtering conditions for a number of different organic materials. Experimental factors include incident ion energy; ion beam angle of incidence; and sample temperature.

[1] D.E. Weibel, N. Lockyer, J.C. Vickerman, App. Surf. Sci., **2004**, *231-232*, 146-152.

[2] A.J. Roberts, S.J. Hutton, C.J. Blomfield, I. Drummond, S.C. Page, *J. Surf. Anal.*, **2009**, *3*, 287.

[3] A. Rafati, M.C. Davies, A.G. Shard, S. Hutton, G. Mishra, M.R. Alexander, *J. Controlled Release*, **2009**, *138*, 40–44

[4] R. Mollers, N. Tuccitto, V. Torrisi, E. Niehuis, A. Licciardello, *Applied Surface Science* **2006**, *252*, 6509-6512.

11:20am **AS-WeM11 The Application of Digital Techniques to the Calibration of Depth Scales in XPS Sputter Profiling**, *P. Mack, T. Nunney, R.G. White,* ***A. Wright***, ThermoFisher Scientific, UK

When sputter profiling a multi-layer material using XPS, it is often difficult to obtain an accurate calibration of the depth scale. In part, this is due to the fact that the sputter rate in each material is different and a post profile measurement of crater depth will only yield an average value for the sputter rate. The purpose of this paper is to provide a method for an internal, standardless calibration of the depth scale which can be applied to single profiles through multilayer materials.

As a layer is etched to a thickness of a few nanometres, the spectrum from the underlying layer can be seen. With knowledge of the electron attenuation lengths in the layers it is then possible to calculate the thickness of the remaining upper layer. If this calculation is carried out following each of a number of sputter cycles then the sputter rate within the layer can be calculated. This value can then be applied to sputtering within the whole of the upper layer. If this is repeated for each layer in a multilayer sample then an accurate depth scale can be constructed.

This method will be applied to a number of samples including standard multilayer materials and the value of the method assessed.

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