Friday Morning, November 19, 2004

Applied Surface Science
Room 210A - Session AS-FrM

FIB and Novel Ion Analysis Methods

Moderator: F.A. Stevie, North Carolina State University

8:20am **AS-FrM1 Elemental and Surface Analysis Via Focused Ion Beam Induced X-Rays**, ***L.A. Giannuzzi,*** FEI Company

Characteristic X-ray emission result from ion/solid interactions, and is the basis for the well known analysis technique referred to as particle induced X-ray emission (PIXE). Characteristic X-rays may be emitted by either bombardment by MeV protons or heavy ions of a few keV. The advantage to heavy ions is that the X-ray yield is confined to a narrow region near the surface. Since the stopping power for < 30 keV Ga+ ions may be orders of magnitude greater than the stopping power for < 30 keV electrons, the acquisition of characteristic X-rays from regions containing both excellent spatial resolution and excellent depth resolution using a focused ion beam (FIB) instrument rather than a scanning electron microscope (SEM) as the primary source are feasible. An additional advantage of heavy ion induced X-ray emission over electron induced X-ray emission is that the Bremsstrahlung is orders of magnitude lower. Thus, ion induced X-ray spectra provides for superior peak to noise ratios, and therefore, offers the possibility for trace element sensitivity compared to electron induced X-ray emission via e.g., X-ray energy dispersive spectrometry (XEDS). In addition, the near surface ion/solid interactions also allow for the possibility of surface analysis via FIB induced X-ray analysis (FIBIX). An added advantage of the FIBIX technique is its sensitivity to soft X-rays, and therefore, light elemental analysis.

8:40am **AS-FrM2 FIB for Materials Characterization, Device Creation and Sample Preparation**, ***R.J. Young,*** FEI Company **INVITED**

Focused ion beam (FIB) systems and DualBeam (combined FIB-SEM) systems have become key tools in the high-resolution characterization of materials, most notably in the semiconductor and data-storage industries, but also extending to many other disciplines where localized sample preparation and analysis is required. Site-specific cross-sections and transmission electron microscope (TEM) samples through disparate materials can be prepared using FIB milling. On a DualBeam the SEM can be used to directly monitor the sample preparation, allowing the section to be precisely positioned relative to a sub-surface feature that is exposed during the sample preparation. In addition, high-resolution, high contrast STEM (scanning transmission electron microscopy) imaging is possible with the electron beam, enabling more problems to be solved in the DualBeam without resorting to the TEM. Ion beam and electron beam induced gas chemistry is also possible, enabling the localized deposition of conductors and insulators, and the selective etching of materials. These capabilities are used in characterization applications for surface protection and delineation of cross-section faces, and also allow integrated circuits to be rewired to debug or prototype the device. Similarly, micro- and nano-scale devices can also be created or modified by the FIB/DualBeam, enabling rapid investigations into novel structures that would be impractical to create by other methods.

9:20am **AS-FrM4 High Spatial Resolution XPS Analysis of Focused Ion Beam Irradiated Specimens**, *J.L. Fenton,* ***K.M. Archuleta,*** *J.E. Fulghum,* The University of New Mexico*; D.P. Adams, M.J. Vasile,* Sandia National Laboratories

Focused ion beams (FIB) are utilized in applications ranging from the preparation of samples for SEM and TEM analysis to machining of micro-tools. Despite their widespread use, there have been few detailed studies identifying how ion bombardment affects the chemistry of the near-surface region. The goal of this project is to assess the impact of Ga+ resulting from FIB preparation. High energy (30 keV) focused ion beam sputtering was first used to mill >100 µm wide features in Si, C and GaAs substrates. Both quantitative, high spatial resolution imaging and spectra-from-images methods were then used to characterize surface chemical distributions. The Ga distribution on the surface was determined in each case, and the impact of implanted gallium on surface oxidation was evaluated. The change in surface stoichiometry with ion dose (from approximately 10@super 15@ - 10@super 18@ ions/cm2) is also discussed. Atomic force microscopy and TEM have been used to investigate the evolution of morphology with ion dose so to aid the interpretation of XPS data.

10:20am **AS-FrM7 Examining the Impacts of Ion Sputtering on Nanoparticles and Nanoporous Materials**, ***A.S. Lea,*** *M.H. Engelhard, D.J. Gaspar, J.R. Williams, D.R. Baer,* Pacific Northwest National Laboratory

Nanostructured materials of various types and forms are increasingly subject to every type of chemical and physical analysis possible. During the course of studies on several different types of 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. To confirm such effects, we need to know many details about size, size distribution, density, and shape that are not always readily obtained. This presentation will review our efforts to quantify the sputter and damage rates for some nanomaterial systems, including iron oxide nanoparticles and porous silica films. We are working to expand our range of information in more details for these materials and to different types of nano-sized objects. Material removal rates are monitored by XPS and AES and material structure information, including material amounts, is also measured by a variety of methods including TEM, XRD and Nuclear Reaction Analysis.

10:40am **AS-FrM8 Determination of Optimum Depth-Resolution Conditions for Time-Of-Flight Medium Energy Backscattering**, ***R.D. Geil,*** *B.R. Rogers, Z. Song,* Vanderbilt University

Measurements of depth resolution in time-of-flight medium energy backscattering analysis have been made in ErAs and ScAs single crystal films on GaAs (100) as a function of depth, beam energy, and analysis angle using He@super +@ as the analysis ion. Film thicknesses ranged from about 5 Å to 50 Å. Experiments were performed with beam energies ranging from 100 keV to 270 keV with the targets oriented at angles ranging from 5° to 55°. Multiple scattering and straggling effects limited depth resolution at analysis angles near 55° while the resolution of the spectrometer was the limiting factor for angles near normal to the beam. Estimates of depth resolution were made from theoretical calculations and were shown to be in good agreement with experimental values when the analysis angles were small. The departure of theoretical values from experimental measurements can be attributed to multiple scattering events and surface roughness.

11:00am **AS-FrM9 Quantitative Profiling of Thin Films by Means of Elastic Recoil Detection Analysis (ERDA) with High Energetic Heavy Ions**, ***W. Bohne,*** *J. Röhrich, E. Strub,* Hahn Meitner Institut Berlin GmbH, Germany **INVITED**

Heavy-Ion ERDA (Elastic Recoil Detection Analysis) can be used to characterize thin solid layers. The concentrations of chemical elements can be determined as well as the layers' thickness and depth profiles. For the measurement, the sample is irradiated with a heavy ion beam. Atoms of the sample are recoiled and detected with an energy and mass dispersive spectrometer. Since the ERDA principle is based on the classical Rutherford scattering theory, the expected yields for the given conditions can be calculated exactly. Therefore, ERDA is a standard-free method. Absolute concentrations can be determined for all detected chemical elements simultaneously, including hydrogen, with almost the same sensitivity. With the time-of-flight ERDA setup at the Hahn-Meitner-Institut (HMI), there are measured almost 500 samples per year. As a standard projectile beam gold ions with an energy of 350 MeV are used. Typical requests are determination of the stoichiometry, in-depth element distributions, concentration of impurities, and the validation or calibration of data from other analytical methods. Typical artifacts or systematic uncertainties of these methods can be ruled out by comparison with the ERDA data. There will be presented a selection of current ERDA measurements mainly concerning the characterization of materials for thin film photovoltaic devices developed at the HMI. Also a comparison with other analytical methods will be shown. The pros and cons of ERDA measurements will be dicussed.

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