QSA-10 Topical Conference
Room Hall A-C - Session QS-TuP
Aspects of Quantitative Surface Analysis

QS-TuP1 Test Studies on Charging, Charge Compensation and XPS Binding Energy Referencing for Al-Ox on Al and SiO@sub 2@, *M.H. Engelhard*, *D.R. Baer, D.H. Kim, D.J. Gaspar,* Pacific Northwest National Laboratory

The ability to determine absolute binding energy remains a challenging problem for X-ray Photoelectron Spectroscopy analysis of insulating or partially insulating specimens. Many studies have demonstrated a variety of difficulties with most approaches. Because our work involves study of many oxide and insulating materials, we have developed a number of approaches for dealing with charge compensation and charge referencing. To extend our understanding of charging in XPS, we have been examining how specific properties of a specimen influence the different approaches to dealing with surface charging. In this paper we add carbon over-layer thickness to measurements that examined the impact oxide layer thickness. Oxidized Al films and SiO2 samples that have been coated with different amounts of evaporated carbon. We have examined the peak widths and apparent binding energies as a function of charge compensation conditions for a Phi Quantum 2000 and Kratos Axis 165 XPS spectrometers. Results assist understanding of the charging process and indicate methods for charge compensation and referencing.

QS-TuP2 A Multi-modal Imaging and Visualization System for 3-D Materials Characterization*, J.E. Fulghum*, *K. Artyushkova, J. Farrar,* University of New Mexico*; L. Broadwater,* Kent State University*; J. Fenton, S. Pylypenko, D. Barbash,* University of New Mexico

Rapid image acquisition has transformed laboratory XPS imaging from a novelty to an increasingly routine analysis method for surface chemical characterization on the scale of microns. A variety of techniques, including FTIR, AFM and confocal microscopy (CM) have fields-of-view which are comparable to imaging XPS, making correlative analyses possible. With appropriate sample marking, information can be acquired from the same area on samples using multiple techniques. Correlating and combining this information allows us to model chemical changes within the sample through visualization techniques. The end result will be a three-dimensional model of the complex chemical structures and morphologies formed in multicomponent, heterogeneous samples. An additional goal is to utilize multivariate analysis methods to extract quantitative data from images and link them to chemical information. Proper integration of useful data from the separate techniques is essential. A comprehensive image analysis system - the Active Knowledge Mesh Model (AKM) - is currently under development in our laboratories. Image analysis involved in AKM has several steps, depending on the properties of the images and prior knowledge of the system and the experiment. Correlating the data from multiple modalities requires experimental matching and marking, image registration, multivariate image analysis, image quantification and image fusion. A prototype interface of AKM architecture involving all these steps will be shown. This work has been partially supported by NSF ALCOM (DMR89-20147), NSF CHE-0113724 and UNM.

QS-TuP3 Measurement of Overlayer Properties Using Angle Resolved XPS, *P. Mack, B. McIntosh,* ***R.G. White,*** Thermo Electron*; J. Wolstenholme,* Thermo Electron, UK

Ultra-thin films (<10 nm) are encountered, for example, as gate oxides on transistors, self-assembled monolayers or surface contamination layers. Angle resolved XPS (ARXPS) can provide a convenient and non-destructive method for measuring thickness and distribution of the component materials within these layers. It will be shown that methods based on sputtering or single angle XPS measurements may produce misleading results. The equation commonly used for the calculation of overlayer thickness will be shown to have limitations that depend upon both the material and the overlayer thickness. Advanced methodologies for the determination of layer thickness from ARXPS data will be described and will be shown to overcome these problems. These methods include the simultaneous calculation of thickness of each layer in a multi-layer stack by fitting. The methods for measuring concentration depth profiles will be discussed. In some applications, it is essential to know the dose of an element within an ultra-thin layer, for example the distribution of nitrogen in a transistor gate oxynitride. It will be shown that large errors can be expected if such measurements are attempted using XPS without knowing the distribution of the nitrogen within the layer. Using ARXPS, the nitrogen distribution can be measured and a more accurate dose calculated from this distribution. Application of ARXPS to thin films will be shown to be a powerful technique for the measurement of dose distribution and thickness but care must be taken to ensure that all of the required parameters are taken into account.

QS-TuP4 Experimental Determinations of Electron Inelastic Mean Free Paths in Ten Elemental Solids from Elastic Peak Intensities, *S. Tanuma,* *T. Kimura,* National Institute for Materials Science, Japan*; K. Goto,* Nagoya Institute of Technology, Japan*; S. Ichimura,* National Institute for Advanced Industral Science, Japan

The values of electron inelastic mean free paths (IMFPs) are very important physical quantity for surface analyses by AES and XPS. In usual, they have been determined from the theoretical calculations because the reliable experimental determinations of IMFP is rather a complicated work. The elastic-peak electron spectroscopy (EPES) is an efficient tool for experimental determination of IMFPs. This method, however, requires a reference specimen to obtain the values of IMFPs and gave different values according to the used reference specimen. On the other hand, absolute elastic-peak measurement method, which is carried out with a novel cylindrical mirror analyzer equipped with a Faraday cup, does not need the reference specimen. However, this method needs the surface plasmon excitation correction. Then, we have measured the IMFPs for Si, Zn, Cu, Ag, Ta, W, Au, Mo, Fe and Pt using Ni reference in the range 50 -2000 eV and compared with the IMFPs determined from theoretical calculations and absolute elastic-peak method using Oswald surface excitation correction. The experimental elastic peak intensity rations of above elemental solids to Ni-reference are in excellent agreement with those of Monte Carlo calculations from TPP-2M and from theoretical IMFPs (Penn algorithm) in the 200 - 5000 eV energy range. Under 200 eV, however, the measured intensity rations of them did not coincide well with the calculated result, which was mainly due to the surface excitation effects and the uncertainty of used elastic scattering cross sections.

QS-TuP6 An Analysis of Rotating-Compensator Ellipsometers for Metrology*, D.E. Aspnes*, *M. Asar,* North Carolina State University*; T. Mori,* Tohoku University, Japan*; K.A. Ebert,* Thermawave, Inc.

Increasing demands on accuracy are moving spectroscopic-ellipsometry technology from rotating-analyzer and -polarizer (RAE, RPE) configurations to rotating-compensator (RCE) designs. The additional 4@omega@t component generated in the transmitted intensity by an RCE makes it intrinsically more powerful for analysis, yet questions concerning relative performance, optimum configurations, and optimal component settings remain. Here, we discuss aspects relevant for achieving maximum accuracy for nm-scale thin-film metrology. First, calculations show that precisions on sample parameters obtained by an RCE are often better than those attainable with an RAE or RPE even for compensator retardations near 180 degrees, thereby resolving the optimum-sensitivity issue. Second, a comparison of RAE and RCE data obtained on a bench system that was operated as an RAE for over 25 years revealed a previously unsuspected polarizer-leakage artifact that affected the RAE data to about 1%, highlighting the more powerful diagnostic capabilities of an RCE. Third, we treat depolarization artifacts in general, using a system-transfer-matrix approach that also allows sample parameters to be extracted from RCE data without having to Fourier analyze the transmitted intensity. Finally, we analyze monoplate and biplate compensators, in particular discussing the multiple-internal-reflection artifacts that lead to the appearance of interference oscillations in spectra obtained with systems using biplate compensators. The optimum ellipsometer configuration is an RCE operating with a monoplate compensator.

QS-TuP7 Characterization of Buried Nanowire by X-ray Standing Wave Method*, A. Saito,* Osaka University and RIKEN harima Institute, Japan*; K. Matoba, J. Maruyama,* Osaka University, Japan*; W. Yashiro,* National Institute of Advanced Industrial Science and Technology, Japan*; T. Ohashi, H. Takaki, K. Takami, M. Akai-Kasaya, Y. Kuwahara,* Osaka University, Japan*; Y. Yoda, O. Sakata,* Japan Synchrotron Radiation Research Institute*; K. Miki,* National Institute for Materials Science, Japan*; M. Aono,* Osaka University, Japan

A perfect bismuth atomic nanowire on a clean Si(001) surface has recently attracted great interests.@footnote 1@ The STM studies showed that this wire is qualitatively different from the other atomic wires that are formed by group-III or -V elements at the initial stage of growth on Si(001): the Bi wire is free of kinks or defects, the width of 1 nm is kept for more than 400 nm long, and the wire appears to be rigidly embedded within the surface, not as adsorbates. Although X-ray photoelectron diffraction and the

theoretical analysis were applied to reveal the Bi wire structure in UHV condition, the wire in the buried interface can be hardly estimated because of the methodological difficulty, despite its importance in application. X-ray standing wave (XSW) method is suitable for this ordered, dilute, and embedded system. This method is based on to excite selectively a specific element and to control the X-ray wave field in the substrate crystal under diffracting condition. Since the spatial controllability of the wave field is precise in about 0.001 nm (0.01Å), the atomic site of the element can be determined quantitatively with respect to the substrate Si lattice plane. In this paper, we present the XSW analysis of the Bi nanowire, where the Bi atomic site was estimated and compared among three different conditions (UHV, amorphous Si cap, epitaxial Si cap). The same analytical method was available for characterization of the low-dimensional system with the different capping layers by use of the third generation synchrotron radiation source. @FootnoteText@@footnote 1@J.H.G.Owen et al.,Phys.Rev.Lett.88,(2002) 226104.

QS-TuP8 Phase Separation at the Surface of PLLA/P104 Blends*, J. Yu*, *J.A. Gardella,* State University of New York at Buffalo

Blends of Pluronic P104, poly(ethylene oxide) (A) poly(propylene oxide) (B) ABA block copolymer surfactant, and PLLA, poly(L-lactic acid), are considered as a delivery matrix for active proteins, nutrients, and essential amino acids in the application as Synthetic Basement Membrane for tracheal wound repair. The amphiphilic block copolymer Pluronic P104 was investigated using X-ray photoelectron (XPS), FTIR and NMR. In blends of different bulk compositions the enrichment of each component was examined by XPS and the thickness of each layer was listed to study the relationship between the bulk composition and surface segregation.

QS-TuP9 Factors Influencing Angle Dependent XPS to Si Oxide Films*, N. Sanada*, *A. Tanaka, A. Yamamoto,* ULVAC-PHI, Inc., Japan

The chemical structure and thickness of an ultra thin film with 1 to 3 nm on a crystalline semiconductor is of interest because its application in semiconductor industries such as gate oxide (including silicon oxy-nitride and high-k gate oxide) thin films and ultra thin passivation films. An XPS measurement is the peculiar nondestructive technique on the chemical in depth analysis of ultra thin films. In addition, XPS has advantages in reproducibility and sensitivity in theory compared with other non-destructive surface analysis techniques. By this means, XPS is anticipated as a tool to evaluate the chemical in depth analysis of the ultra thin films, especially in a measure of the industrial demand. We have gauged several latent variables in XPS measurements, for examples, photoelectron diffraction of crystalline silicon substrate, acceptance angle of the photoelectron analyzer, X-ray probe stability, and damages in the XPS measurement. Those issues are evaluated as a matter of fluctuation of the film thickness. The photoelectron diffraction of the substrate silicon is the heavy factor to fluctuate the analysis results. A commercial XPS instrument which has an acceptance angle with 4 degree in polar angle and 20 degree in azimuthal angles, even has 6% fluctuation in Si 2p photoelectron intensity of the substrate silicon at 45 degree in polar angle, which is confirmed by an experiment and a calculation. On the other hand, XPS measurement is evident to be reliable by avoiding the particular measurement angle and using an instrument with a wide acceptance in azimuthal angle. Finally, we applied the angle dependent XPS analysis to two-layered high-k gate oxide thin film with identical constituent elements for both layers. Those results will be discussed in detail.

QS-TuP10 Comparison of Model Predictions and Experimental Measurements of Linescans Across an Interface by Auger Electron Spectroscopy*, S.A. Wight*, *C.J. Powell,* National Institute of Standards and Technology

In order to determine the lateral resolution in scanning Auger microscopy of a practical sample, measurements and simulations have been made of variations in Auger signals across a selected interface. The interface for these experiments consisted of a selected line of chromium oxide over chromium on an indium-tin oxide coated quartz substrate. Auger electron spectra were collected between 300 eV and 600 eV as the beam was stepped across the interface in a direction perpendicular to the interface. The spectra at each point in the linescan were then processed to extract the relative contributions of the spectra for the two materials measured far from the interface. The normalized data were plotted against beam position on the sample to visualize the sharpness of the interface and to obtain a measure of the lateral resolution for the Auger signals. This procedure was repeated for multiple accelerating voltages of the primary electron beam. The experimental linescans were compared with simulated linescans for an ideal interface based on a simple model that represents contributions to the Auger signal from ionizations caused by the primary electrons and backscattered electrons. Preliminary results show qualitative agreement. We also show that the radius of the analysis area varies appreciably with parameters describing the Auger signal due to backscattered electrons.

QS-TuP11 Quantitative Image Analysis of Low-energy Electron Diffraction Patterns to Obtain Surface Geometries of Amines Adsorbed on the Si(100)-(2x1) Surface*, J.K. Dogbe*, *S.M. Casey,* University of Nevada, Reno

Experimental and computational low-energy electron diffraction (LEED) intensity vs. voltage (IV) curves were used to analyze the surface geometries of amines adsorbed on the Si(100)-(2x1) surface. Both the clean silicon surface and the ammonia-covered surface were used as calibration systems. For silicon, the LEED IV curves obtained agree well with literature data to within experimental uncertainty. The results for ammonia adsorption are compared to results from recent photoelectron diffraction studies of this surface. Results of LEED IV probing of methylamine adsorption will be presented and compared to computational treatments of probable reaction pathways for this class of molecule on silicon.

QS-TuP12 Physically Measuring Thickness' of Thin Films via Atomic Force Microscopy: Design and Use of The Abruptor*, G. Acosta,* ***D. Allred****, R. Davis,* Brigham Young University

In our research of thin films (typically our films are 2.5-15 nm thick), we invest a great deal of time and energy in characterizing our films' physical and optical properties. We have found that it is imperative to have the ability to determine, with confidence, the thickness of our films when we try to describe a material's optical performance, especially true for work in the extreme ultraviolet. Unfortunately, we've learned this is not as simple a task as it would seem. Methods that are optical in nature used to determine how thick a film become more difficult to employ and less effective for very thin films. We would much rather be able to use an AFM to physically measure a film's thickness. Having spent two years refining such a technique, we are happy to report the method we have developed. Our technique involves using a rigidly supported, stainless steel razor blade to mask the substrate during deposition, leaving a distinct, abrupt edge when removed. The device is named the Abruptor. In addition to now being able to determine the thickness of our films, we also offer a side note regarding hydrocarbon contamination of substrates (on the sub-nanometer scale) from exposure to ambient.

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