QSA-10 Topical Conference
Room 320 - Session QS-MoM
Advances in Quantitative Surface Analysis

Moderator: C.J. Powell, National Institute of Standards and Technology

8:20am **QS-MoM1 Modules for an XPS Expert System: Applications in Corrosion Science*, J.E. Castle,*** University of Surrey, UK **INVITED**

Expert Systems (ES) can guide users in application of XPS.@footnote 1-6@ Guidance requires modules of the ES to be tailored to a particular group, e.g., corrosion scientists. This group, which has defined needs and a history of using XPS, could benefit from automated knowledge retrieval. In creating a set of rules, the requirements are defined and matched to the information that might be delivered by XPS, e.g.: the mean surface composition and relation to the bulk; the location of any marker elements; the thickness of overlayers, e.g., a contamination layer and an oxide or similar layer; evidence for graded composition such as enrichment of an alloying element in the oxide or metallic undersurface; valance states and a trial stoichiometric composition. Reminders of further opportunities should also be provided, possibly by use of on-screen wizards.@footnote 4,5@ In proposing this ES module emphasis will be placed on the cost-effective provision of basic information from the 'survey scan'. The rules have provenance from prior discussion and publication by international workshops such as those sponsored by IUVSTA.@footnote 1,3,6@ The rule base will be most useful when it is embedded in the controlling system for the spectrometer, so that some degree of knowledge based acquisition is possible, but would be equally suitable for stand alone, retrospective analysis. Both outcomes make a demand on costly resources that in the end will have to be provided by the user community. It is hoped that this feasibility study will show that, for corrosion scientists, this is worthwhile. @FootnoteText@ @footnote 1@M-G Barthes-Labrousse, Surf.Interface Anal. 26 p72 - 91 (1998) @footnote 2@J.E.Castle and M.A.Baker, J. Elec Spec Rel. Phenom., 105 p245 - 256 (1999) @footnote 3@L.Kover, Surf. Interface Anal. 29 p671 - 716 (2000) @footnote 4@J. E. Castle, PV 2001 - 5, C.R.Clayton et al. Eds. The Electrochemical Soc., NJ. pp1-14(2001) @footnote 5@J.E.Castle, Surf.Interface Anal. 33 pp 196 - 202 (2002) @footnote 6@ Report of St Malo Workshop, to be pub.

9:20am **QS-MoM4 Auger Electron Spectroscopy: Reducing Measurement Uncertainty*, J.D. Geller,*** Geller MicroAnalytical Laboratory **INVITED**

Quantitative analysis using Auger electron spectroscopy (AES) is most commonly performed using pure element sensitivity factors. The accuracy of the technique can be quite good for materials such as stainless steels where many of the elements have adjacent atomic numbers and similar densities. However, in most other materials errors well over 100% of the accepted true value are commonplace. The reasons for this will be discussed and different methods for quantifying data will be compared for their effectiveness. Accuracy, defined as the difference between the accepted true value and the result of an analysis, with Auger electron spectroscopy has seen very slow improvements over the years. There have been relatively very few technical papers addressing this important subject. By contrast, highly accurate quantitative analysis with the electron probe micro analyzer (EPMA) developed very quickly to the point where accuracies in the range of a few percent on a relative basis are commonly reached. Programs for converting x-ray intensities to concentrations following the ZAF (where Z is atomic number correction, A the absorbance correction, and F the fluorescence correction) procedures are available both in the public domain as well as from all the manufacturers of x-ray spectroscopic equipment. For AES there are no commercially available programs beyond the sensitivity factor approach. This work evaluates the current matrix effect corrections approaches (Sekine, et al) to that using elemental sensitivity factors from measurements with compounds. Sensitivity factors for several experimentally collected stoichiometric carbides, silicides, phosphides and sulfides are compared with those calculated from pure element sensitivity factors using matrix effect corrections. The factor with the greatest uncertainty was found to be preferential ion sputtering which is not included in any of the corrections. Sekine, et al, Evaluation of Correction Accuracy of Several Schemes for AES Matrix Effect Corrections, Surface and Interface Analysis, Vol. 15, 466-472 (1990).

10:20am **QS-MoM7 Molecular Characterization of Biomaterials with ToF-SIMS Imaging*, N. Winograd,*** Penn State University **INVITED**

It is now feasible to employ ion beams to desorb molecules directly from a variety of complex matrices. Here we show that it is possible to examine frozen-hydrated biological interfaces prepared using a special freeze-fracture protocol. The results demonstrate that it is possible to unravel dynamical events such as chemical fluctuations associated with domain structure in cellular membranes. This strategy allows the distribution of molecules within these matrices to be determined with high lateral resolution. Fracturing criteria for preserving chemical distributions have proven to be much more stringent than morphological cryo-electron microscopy studies. Two examples will be discussed. In the first instance, we report on the characterization of liposome particles that are about 50 microns in diameter. We have captured each stage of a fusion event between two liposomes and have shown that membrane structure during fusion ranges from specific domains that then migrate across the interface to produce a homogeneous, fluid-mosaic membrane. In the second instance, several different possible fracture planes from single biological cells have been imaged to understand further the process of freeze fracture and TOF-SIMS imaging of cellular membranes. Sensitivity is still problematic for this type of mass spectrometry. We present recent imaging data using a C60+ ion source that increases the yield of biomolecules by more than a factor of 1000. Combined with the freeze-fracture protocols, this technology holds the promise to define the biology of cellular membrane surfaces at the molecular level.

11:20am **QS-MoM10 Extrinsic and Intrinsic Excitations in Ge and Si Photoelectron Spectra*, L. Kövér,*** *Z. Berényi,* Institute of Nuclear Research of the Hungarian Academy of Sciences, Hungary*; S. Tougaard,* University of Southern Denmark, Denmark*; F. Yubero,* Institutio de Ciencia de Materiales de Sevilla, Spain*; I. Cserny, J. Végh, J. Tóth, D. Varga,* Institute of Nuclear Research of the Hungarian Academy of Sciences, Hungary*; W. Drube, T. Eickhoff,* HASYLAB at Deutsches Elektronen-Synchrotron DESY, Germany

Studying excitations accompanying deep core photoemission and Auger transitions in solids of great practical interest is important for understanding solid state effects@footnote 1@ on such transitions and for revealing the electronic structure of these systems. Here we report our results for (Ge 1s,2s; Si 1s) core photoelectron spectra excited by X-rays of different energies. Photoelectron spectra were obtained using the Tunable High Energy XPS facility at the BW2 beamline of HASYLAB,@footnote 2@ while the electron energy loss spectra reflecting the effects due to inelastic scattering (extrinsic losses) were measured with the home built ESA-31 electron spectrometer.@footnote 3@ The background corrected spectra are compared to model spectra calculated within the frame of a theory@footnote 4@ developed to describe both extrinsic and intrinsic (due to the appearance of the core hole) losses in XPS experiments. In addition, the ratio of the probabilities of extrinsic and intrinsic plasmon creation has been derived analyzing the excited plasmon spectra and is compared to the ratio obtained from the theory and from an other experimental approach. In a reasonable agreement with the theory, our analysis of the measured spectra shows a significant contribution from intrinsic excitations. Supported by OTKA T038016 and IHP-Contract HPRI-CT-1999-00040/2001-00140 (EC). @FootnoteText@ @footnote 1@ L. Kövér, Zs. Kovács,J. Tóth, I. Cserny, D. Varga, P. Weightman and S. Thurgate, Surf. Sci., 433-435(1999)833.@footnote 2@ W. Drube, T. M. Grehk,R. Treusch and G. Materlik, J. Electron Spectrosc. Relat. Phenom.,88-91(1998)683. @footnote 3@ L. Kövér, D. Varga,I. Cserny, J. Tóth and K. Tökési, Surf. Interface Anal., 19(1992)9. @foonote 4@ A. Cohen-Simonsen, F. Yubero and S. Tougaard, Phys. Rev. B56(1997)1612.

11:40am **QS-MoM11 Multivariate Analysis for XPS Spectral Imaging@footnote 1@*, D.E. Peebles,*** *J.A. Ohlhausen, P.G. Kotula,* Sandia National Laboratories

The acquisition of complete spectral images for x-ray photoelectron spectroscopy (XPS) is a relatively new approach, although it has been used with other analytical spectroscopy tools for some time. This technique generates full spectral information at every pixel of an image, in order to provide a complete chemical mapping of the imaged surface area. Multivariate statistical analysis techniques applied to the spectral image data provide a way to sort through this large block of data to determine the chemical component species present as well as their distribution and concentrations, with minimal data acquisition and processing times. The benefits of multivariate analysis of the spectral image data include significantly improved signal to noise and improved spatial resolution, which are achieved due to the large number of data points included in the

image. In this paper, we will illustrate the signal to noise and spatial resolution obtained from a Magnification Reference Standard at a series of spectral image acquisition times, with a direct comparison of the raw images to the multivariate processed data. We will demonstrate the elemental separation and chemical discrimination possible with Sandia's novel multivariate statistical analysis approach for both limited spectral region acquisition as well as more complete spectral imaging data sets. It will be shown that Sandia's techniques provide efficient methods for deriving physically realistic chemical components without user input other than the spectral data matrix itself. @FootnoteText@ @footnote 1@ This work was completed at Sandia National Laboratories, a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

QSA-10 Topical Conference
Room 320 - Session QS-MoA

Thin-Film Metrology

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

2:00pm **QS-MoA1 Metrology Needs for Ultrathin Films in the Wafer Processing Industry. What is Needed? What is Available?*, C.R. Brundle,*** C.R. Brundle and Associates*; C.A. Evans,* Evans FWA **INVITED**

Single layer films of less than 1nm (eg Si/O/N) or stacks of less than 4nm (eg TaN/Cu) are already in production or under development. The ITRS required metrology for these, however, has been lagging because this presents major instrumentation challenges. Thickness SPC specs across wafers and from wafer to wafer are typically 3% RSD, or better, which implies a measurement precision (at 1 sigma) of at least 1%, ie 0.01nm for a 1nm film. Elemental composition precision may need to be similarly tight (eg for N concentration in a 1nm Si/O/N film). More esoteric parameters, such as depth distribution (elemental or even chemical), interface mixing, roughness, or reaction, or surface contamination, may also have to be under SPC, but even if they do not, these variables must not compromise the metrology used. Finally there may be requirement to make such measurements on restricted areas on production wafers. In this paper we review the capabilities of some of the analytical technologies beginning to find their way into the industry to deal with these issues. They include such methods as VUV spectroscopic ellipsometry, XRR/XRF and XRR/ellipsometry combinations, acoustic sonar methods, non-contact electrical methods, Low Energy X-Ray Emission Spectroscopy (LEXES), and XPS and ARXPS. Examples of their use are presented. It is natural that new measurement instrumentation should penetrate the the "lab" first, where the requirement for throughput and non-expert, recipe driven, automation of both data collection and processing are far less demanding than in "the fab", but even in the lab the instrumentation must must handle full 300mm wafers in a manner where the "time to answer" is acceptable. We discuss what attributes are likely to lead to a transition to fab use.

3:00pm **QS-MoA4 Intercomparison of Silicon Dioxide Thickness Measurements Made by Multiple Techniques - The Route to Accuracy*, M.P. Seah,*** National Physical Laboratory, UK **INVITED**

A pilot project has been launched under the auspices of the Consultative Committee for Amount of Substance (CCQM) to evaluate the measurement issues for the amount of SiO@sub 2@ on (100) and (111) Si in the thickness range 1.5 nm to 8 nm. Wafers and methodologies have been carefully prepared to achieve the best results possible. 44 sets of measurements have been made in different laboratories using 9 different methods (MEIS, NRA, RBS, SIMS, XPS, ellipsometry, GIXRR, NR and TEM). The results have been assessed, against NPL XPS data, using d(respondee) = m d(NPL) + c. All show excellent linearity, except 3 sets with methods more suited to composition depth profiles. The main sets correlate with the NPL data with average rms scatters of 0.15 nm with half being better than 0.1 nm. Each set allows the relative scaling constant, m, and the zero thickness offset, c to be determined. Each method has 0 < c < 1 nm and it is these offsets, measured here for the first time, that have caused many problems in the past. Each technique has a different accuracy for m and consistent results have been achieved. XPS has poor accuracy for m but a high precision and, critically, has zero offset if used correctly. Achieving a consistent scaling constant and zero offset for XPS requires reference conditions and is not trivial. Analysts using these conditions generated dramatically improved data. A combination of XPS and another method allows an accurate determination of the XPS scaling constant. XPS then has a high accuracy, traceable via the other method. Several methods have small offsets which, if they can be controlled, will enable these methods also to show high accuracy.

4:00pm **QS-MoA7 Comparative Thickness Measurements of SiO@sub2@/Si Films for Thicknesses less than 10 nm*, T. Jach,*** *J.A. Dura, N.V. Nguyen, J. Swider,* National Institute of Standards and Technology*; G. Cappello,* Institute Curie, France*; C. Richter,* National Institute of Standards and Technology

The metrology of gate dielectric thicknesses using different methods for layers below 10 nm is still subject to uncertainties. We report on a comparative measurement of SiO@sub2@/Si dielectric film thickness (t<10 nm) using grazing incidence x-ray photoelectron spectroscopy, neutron reflectometry, and spectroscopic ellipsometry. Samples with nominal thicknesses of 3 nm - 7 nm were characterized by XPS with grazing incidence x-rays at 1.8 keV, with cold neutron reflectometry (@lambda@=0.475 nm), and with spectroscopic ellipsometry over 1.5 eV<E<6.0 eV. The results show good agreement between the ellipsometry and the grazing incidence XPS, with slightly lower values for the neutron reflectometry. The role of sample preparation and the assumptions regarding surface contamination are discussed.

4:20pm **QS-MoA8 The Thickness and Composition of Ultra-thin SiO@sub 2@ Layers on Si*, C. Van der Marel,*** *M.A. Verheijen, Y. Tamminga,* Philips Electronics, The Netherlands*; R.H.W. Pijnenburg,* Technical University Eindhoven, The Netherlands*; N. Tombros,* State University of Groningen, The Netherlands*; F. Cubaynes,* Philips Research, IMEC, Belgium

Ultra-thin SiO2-layers are of importance for the semiconductor industry. One of the techniques that can be used to determine the chemical composition and thickness of this type of layers is XPS (X-ray Photoelectron Spectroscopy). As shown by Seah and Spencer,@footnote 1@ it is not trivial to characterize this type of layers in a reliable way. We carried out a series of systematic investigations on layers of SiO2 on Si (in the range from 0.3 to 3 nm). The samples were analyzed by means of TEM, RBS and XPS. The XPS-results were analyzed using the standard Cumpson formula (equation 2 in@footnote 1@), by means of Quases-Tougaard and using an overlayer-substrate model. We also examined the influence of various experimental parameters upon the results (e.g. irradiation time in RBS, objectivity of layer thickness determination in TEM, measuring time and pass energy in XPS). It was found that the ratio O:Si of the layers always corresponds to that of pure SiO2. Yet, for thickness below 2 nm, small but significant deviations were found between RBS and XPS on the one hand and TEM-results on the other hand. The results suggest that the density of SiO2-layers with a thickness below 2 nm is less than the density of bulk SiO2. @FootnoteText@@footnote 1@ M.P. Seah and S.J. Spencer, Surface and Interface Analysis 33 (2002) 640.

4:40pm **QS-MoA9 Characterization and Metrology for High k Materials using Parallel Angular Resolved XPS (PARXPS)*, G. Conti,*** *C.C. Wang, Y. Uritsky,* Applied Materials, Inc.*; C.R. Brundle,* C.R. Brundle and Associates

Owing to the excellent matching of the probing depth of XPS to the current ultrathin film thickness range in use in the wafer processing industry (0.5 to 4 nm), this technique is coming to the forefront for providing composition and chemistry information, both in the film and at the surfaces and interfaces. Ideally one would like a technique where a protocol for measuring thickness, composition, and a chemical concentration depth profile can be measured non-destructively. XPS can do this by exploiting PAR-XPS to obtain a depth profile, as opposed to the traditional sputter profile approach, which is both destructive and often introduces artifacts. In PAR-XPS there is no need to rotate the wafer to get series of angular resolved spectra, since it is collected simultaneously over a wide angular range and binned into user defined smaller angular ranges. We present examples of thickness, N dose, and N depth profile characterization for Si/O/N gate oxide (1nm to 3nm range), and for HfO2 films on SiO2 on Si (3nm to 4nm total range) using the VG Theta Probe 300. For the Si/O/N films we show that high precision (better than 1% RSD) is obtainable for thickness and apparent N dose using an integrated angle (22 to 63 degrees) measurement. To turn the apparent N dose into a true dose requires information on the N depth profile, which is obtained from the angle resolved measurements, using the Maximum Entropy modeling approach. For the HfO2/SiO2/Si films the PAR-XPS shows that the films are intermixed for the particular processing conditions discussed here. Our conclusions on the use of PAR-XPS are that the method can meet the needs for precise film thickness measurements, and depth resolved chemistry, provided the film thickness to be analyzed is less than about 4nm. Beyond that, traditional spattering becomes necessary, since PAR-XPS contains too small a component of information from such depths.

5:00pm **QS-MoA10 Multivariate Statistical Analysis of Spatially Compressed Time-of-Flight Secondary Ion Mass Spectrometry Images@footnote 1@*, J.A. Ohlhausen,*** *M.R. Keenan, P.G. Kotula, D.E. Peebles,* Sandia National Laboratories

Owing to the parallel nature of Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS), complex and very large datasets can easily be acquired. An example of such a large dataset is a spectral image where a complete spectrum is collected for each pixel. Ideally, the complete spectral image would be used to provide a comprehensive materials characterization. This is difficult to accomplish with traditional techniques. Previously, we have demonstrated the application of multivariate spectrum imaging techniques to TOF-SIMS. This technique, called Automated eXpert Spectral Image (and series) Analysis -- AXSIA, is based on the separation of

a complex and very large spectral image dataset into physically realizable and intuitive chemical components, including both spectra and concentrations. The full analysis is performed without outside estimates of spectral shapes, concentrations or the number of components present. In TOF-SIMS, we have shown that spectral series in the form of depth profiles (1D), images (2D), and imaged depth profiles (3D) can be analyzed using AXSIA. Since datasets can be large (5MB-1GB), data compression must be performed in order to process the data on laboratory computers. While providing signal-to-noise and memory storage improvements, data compression can hide or dilute important and small features. In this talk, I will present some statistical advantages of using multivariate techniques directly to spatially compressed data while maintaining full image resolution. In addition, I will explore the trade-offs between spatial and spectral compression and small feature recognition. @FootnoteText@ @footnote 1@This work was completed at Sandia National Laboratories, a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

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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