## **Tuesday Morning, November 3, 1998**

**Applied Surface Science Division** 

### Room 307 - Session AS-TuM

### Sample Preparation and Tricks of the Trade

Moderator: D.R. Baer, Pacific Northwest National Laboratory

8:20am AS-TuM1 Using a Focused Beam XPS System for Analysis of Oxides, Insulators and Beam Sensitive Materials@footnote 1@, M.H. Engelhard, L.-Q. Wang, B.J. Tarasevich, D.R. Baer, Pacific Northwest National Laboratory

Experience gained during use of a Physical Electronics Quantum 2000 XPS system for the analysis of some oxides, insulators and beam sensitive materials is reported. This instrument, which is part of a Department of Energy user facility, uses an internal monochromatic focused x-ray beam that can be focused, raster scanned, and changed in intensity. Some of our initial studies with this instrument involved comparison of data from the Quantum with measurements made on older systems. These comparisons included an examination of line widths, x-ray damage, and effectiveness of the neutralization methods. In addition to the normal Quantum specimen handling system, the spectrometer can interface with a special specimen handling system that allows interchange among 15 different locations in the user facility. This extra capability allows conduct of a variety of experiments (involving heating, film deposition, electrochemistry or corrosion) but introduces a variety of challenges for specimen mounting. Specific data to be reported include measurements of line-width and the ability to observe defects on the rutile (110) surface and damage observed during analysis of self assembled monolayer and polymer systems. This system now includes the newly developed ion-neutralization capability (U. S. patent 5432345). Because this neutralization system includes the use of both an electron gun and low energy Ar ions, a test was made to determine if the neutralization method introduced defects on a "defect free" rutile surface. No introduction of defects was observed for the period of our test. @FootnoteText@ @footnote 1@This research was conducted at the William R. Wiley Environmental Molecular Sciences Laboratory (EMSL) with funding provided by the U.S. Department of Energy, Office of Basic Energy Sciences. The EMSL is a new DOE scientific user facility located at the Pacific Northwest National Laboratory (PNNL) in Richland, Washington. PNNL is operated by Battelle for the Department of Energy.

### 8:40am AS-TuM2 Implementation and Application of a Ag L@sub alpha@ Monochromatic Source on a Magnetic Lens Based Spectrometer, C.J. Blomfield, B.J. Tielsch, S.P. Page, Kratos Analytical Ltd, United Kingdom

High energy Ag L@sub alpha@ X-rays have several advantages over the more commonly employed AI K@sub alpha@ source. A photon energy of 2984.3 eV means that a Ag source can generate higher core levels, Auger series and has a greater excitation volume. A further advantage over other high energy sources is that the Bragg condition may be satisfied via a second order diffraction within the confines of the conventional AI K@sub alpha@ monochromator body with only minor modifications. Such an approach has previously resulted in a workable source although the sensitivity was reduced in comparison to the Al K@sub alpha @source.@footnote 1@ We have now for the first time implemented a Ag monochromator as a modification to the standard Al mono on a modern magnetic lens based electron spectrometer, the Axis Ultra. The great improvements gained in photoelectron collection efficiency by employing magnetic lens technology improves the sensitivity and ultimate usability of the Ag source making it a viable alternative for the measurement of deep 1s core levels and Auger parameters. The functionality will be demonstrated with examples of Auger parameter and high energy core level spectroscopy. @FootnoteText@ @Footnote 1@K Yates RH West Surface and Interface Analysis Vol 5 No 4 1983 133-138

### 9:00am AS-TuM3 The Good and the Bad About XPS Peak Fitting, N.H. Turner, Naval Research Laboratory

Fitting XPS peaks is done to identify different elemental components or atomic sublevels in cases where there is a complex peak shape. Also, fitting is performed to determine parameters such as peak position, height, and FWHM even for a simple, one component line. Usually Gaussian or Lorentzian line shapes (or a combination of these functions) are used for fitting purposes. Often from these determinations relative atomic amounts of the detected species are computed for the sample being studied. There are many factors that contribute to an observed peak, and a blind acceptance using curve fitting potentially can lead to erroneous interpretations of the experimental data. These mistakes can include physically unrealistic situations and the number and types of components present. In this presentation methods to better use curve fitting will be presented to minimize many of the possible problems. These practical approaches include considering the chemistry and physics of the system being studied, employing reasonable fitting criteria where appropriate, e.g., peak widths and energy separations, relative atomic area ratios, number of variables, curve types, other elements present, and the experimental conditions under which the data were obtained.

#### 9:20am AS-TuM4 Auger Depth Profiling at Extreme Low Ion Energy, *M. Menyhard*, *A. Barna*, *A. Sulyok*, Research Institute for Technical Physics and Materials Science, Hungary

To obtain good depth resolution the sputtering induced surface roughening as well as the ion mixing should be reduced. It is well known that applying Zalar rotation with grazing angle of incidence the surface roughness is considerably reduced and the depth resolution is mainly determined by ion mixing. We have shown previously that the ion mixing proportional to the square root of the ion energy, in the energy range of 4-0.25 keV, thus the best depth resolution can be obtained by applying the lowest possible ion energy. To improve further the depth resolution we have constructed a new ion gun operating down to 0.1 keV. We will report on the experimental findings using this extreme low ion energy for depth profiling, and a novel evaluation procedure (based on dynamic TRIM simulation considering the experimentally determined roughness values ) of the depth profiles. We will show that the sputtering induced surface roughness (specimen is rotated during sputtering) depends on the ion energy; e.g. in case of GaAs we have found 1 nm and non roughness for 1 keV and 0.25 keV ion energy, respectively [1]. We will also show that the square root of ion energy dependence is also valid for this 0.25 - 0.1 keV ion energy range. We will demonstrate the capability of the method on several examples. E.g. we will show that using extreme low ion energy and our novel evaluation method for the depth profiling of a GMR structure, consisting of 1 nm thick Co/Cu layer, the demixing of Cu and Co can be demonstrated. This work was supported by grant OTKA 15880. 1. A. Barna, B. Pécz and M. Menyhard, Ultramicroscopy 70 (1998) 161-171

### 9:40am AS-TuM5 Sample Preparation and Practical Surface (and Interface) AnalysisTricks of the Trade, *D.F. Reich*, Physical Electronics INVITED

In applied surface science, instruments are now available with 'pushbutton' tuning of spectrometers and primary beam columns, automated analysis capabilities, and so on. Nevertheless, far from becoming less important to the analytical process, the analyst remains as critical as ever to the success of the whole enterprise. The aim of this presentation is to discuss some 'practical' knowledge, particularly in relation to sample preparation and instrument operation for surface analysis. My own area of practice is dynamic and static SIMS, using quad and time of flight SIMS instruments. However, the general sample preparation requirements for SIMS are equally encountered in AES, XPS etc. The practical methods discussed should hopefully be of interest to all, not just SIMS practitioners. (1) The following types of sample preparation will be addressed: Cryopreparation of volatile and organic materials: freeze-drying, cryomicrotoming. Cold introduction and cold stages. X-section preparations: fracture methods, microtoming, 'clean' polishing. Mounting of awkward geometry samples: powders, tubes, rods, fibers: woven; strands; loose mats, etc. Solvent cleaning: which solvents to use in the event of contamination, either on samples or on contaminated sample holders. Potential problems for polymers and organic surface treatments. Use of adhesives for sample mounting: forbidden territory? Which products are acceptable and which are not. (2) Certain instrumental parameters will also be addressed: Primary beam probe size effects: flux densities and beam damage. Requirements for charge compensation on insulators. Tricks of the trade. Practical limits. Spectrometer issues: the importance and influence of the energy window of whatever spectrometer (electron or ion) is in use. In summary, the presentation will aim to give you, a fellow analyst, some ideas that may be of use in increasing that part of your knowledge of surface science that is 'practical'.

# 10:20am AS-TuM7 Physical Influences on Chemical Identification using TOF-SIMS, *T.J. Schuerlein*, *G.S. Strossman*, *K.J. Wu*, *T.F. Fister*, Charles Evans & Associates

Time of flight secondary ion mass spectrometry (TOF-SIMS) is rapidly becoming a standard tool for failure analysis and identification of surface contamination. One requirement of a tool used for these purposes is to be able to use reference spectra to help identify unknowns, as is typically done in other techniques such as FTIR. Although TOF-SIMS has had great success in addressing such identification issues, there are some possible pitfalls that are to be avoided when acquiring reference spectra and

# **Tuesday Morning, November 3, 1998**

making subsequent comparisons to analytical data. We have observed a series of physical parameters that can alter mass spectra that are not related to the chemical nature of the analyte. In some cases these effects are subtle, in others the changes observed in the mass spectrum are significant. Data will be shown which illustrates these effects for physical parameters such as contaminant thickness, sample temperature and the interaction between surface species. We will also demonstrate how the intentional introduction of a reagent can be used to increase the molecular ion yield of high molecular weight species.

#### 10:40am **AS-TuM8 SEM Sample Preparation Using Ion Sputtering**, *J.R. Kingsley*, *X. Lu*, Charles Evans & Associates

Wet chemical etching has long been the preferred method for the delineation of features in cross sections of Integrated Circuits. Dry chemical etching, or plasma etching, has also been used as an effective tool for the selective removal of material. One limitation of these techniques is a lack of reproducibility due to such factors as wet etch age, temperature, etch time and composition. Plasma condition changes due to the size and number of samples, the other materials present, and the long time stability of the plasma adversely affect dry chemical methods. In this paper we characterize the use of focused ions, in combination with electron microscopy, to circumvent the inherent inconsistent results noted above. By using focused ions as the etching source in the same vacuum as the imaging source, a reproducible stop point can hopefully be obtained.

11:00am AS-TuM9 The Correlation Between Ion Beam/Material Interactions and Practical FIB Specimen Preparation, B.I. Prenitzer, L.A. Giannuzzi, University of Central Florida; S.R. Brown, Cirent Semiconductor; T.L. Shofner, Bartech Group; R.B. Irwin, F.A. Stevie, Cirent Semiconductor Nanometer scale, high resolution Ga@super+@ ion probes, attainable in commercially available focused ion beam (FIB) instruments, allow sputtering/deposition operations to be performed with a high degree of spatial precision. In addition to semicondutor applications, FIB methods have been applied to the preparation of SEM and TEM specimens from a host of materials that have traditionally proven to be challenging from the standpoint of either composition or geometry. As FIB applications increase in diversity, it becomes necessary to examine the interrelationships between target material, variable processing parameters, and process efficiency of the milling phenomena. The roles of incident ion attack angle, beam current, raster pattern, and target material dependent removal rate are considered as applied to the FIB lift-out method. Careful characterization of such relationships is used to explain observed phenomena and predict expected milling behaviors, thus expediting the fine tuning process for new or novel applications and allowing the FIB to be used more efficiently with reproducible results. Applications involving fibers, powders, and interfaces in metal, ceramic, and biological materials are presented.

# 11:20am AS-TuM10 Use of Micro-Craters and Extended Rotational Profiling for Auger Analysis of Difficult Samples, *R.E. Davis*, IBM Corporation, East Fishkill Facility

When designing, evaluating or operating an Auger electron spectrometer, it is common and appropriate to turn ones attention first to the performance of the primary excitation, and second to the spectrometer. However, in certain types of difficult samples, the desired experimental outcome depends in many cases more on other factors. This paper will describe several examples of such difficult problems which were successfully analyzed by focusing on the sputtering process and the ion gun, with only ordinary emphasis on the electron column and spectrometer. The first example overcomes a common problem for Auger analysts, dealing with very small electrically conductive features that are surrounded by insulating material. With these samples one can make good use of the higher brightness of a magnetically-confined ion source, or duoplasmatron, to depth profile with very small sputtered areas, on the order of ten microns across. Examples of the application of this technique will be drawn from studies of semiconductor technology. The second application involves very thick multilayered stacks of metals, with a thin (200Å) but crucial adhesion layer of chromium buried under seven microns of gold, nickel and copper. By attacking the buried layer from the top, one can establish a baseline for how much oxygen and carbon are present at the bottom of the chromium layer without exposure to ambient, even for films with excellent adhesion. Another example of extended depth profiling will be presented which involved subtle changes in the distribution of @theta@-phase aluminum copper within Al-2% Cu semiconductor interconnects, which had important ramifications in terms of chemicalmechanical polishing.

11:40am AS-TuM11 Two-Dimensional Surface Roughness Measurements of Sidewalls of High Aspect Ratio Patterns Using the Atomic Force Microscope, K.-J. Chao, R.J. Plano, J.R. Kingsley, Charles Evans & Associates Methods of measuring the surface roughness of the sidewalls of high aspect ratio patterns are presented. Cleaving developed resist and etched silicon samples parallel to the long direction of the patterns and tipping over the sample 90 degrees fully exposes the sidewall surfaces, allowing investigation by either the Scanning Electron Microscope (SEM) or the Atomic Force Microscope (AFM). Another method, simply tipping over the lines in the developed resist samples, also allows full access to the resist sidewall. While the SEM can be used to confirm the sidewall surface features, the AFM provides quantitative information such as the Root-Mean-Square (RMS) roughness, unobtainable through other methods.

### **Author Index**

## Bold page numbers indicate presenter

 $\begin{array}{c} - B - \\ Baer, D.R.: AS-TuM1, 1 \\ Barna, A.: AS-TuM4, 1 \\ Blomfield, C.J.: AS-TuM2, 1 \\ Brown, S.R.: AS-TuM9, 2 \\ - C - \\ Chao, K.-J.: AS-TuM11, 2 \\ - D - \\ Davis, R.E.: AS-TuM10, 2 \\ - E - \\ Engelhard, M.H.: AS-TuM1, 1 \\ - F - \\ Fister, T.F.: AS-TuM7, 1 \\ - G - \\ Giannuzzi, L.A.: AS-TuM9, 2 \\ \end{array}$ 

- I --Irwin, R.B.: AS-TuM9, 2 - K --Kingsley, J.R.: AS-TuM11, 2; AS-TuM8, 2 - L --Lu, X.: AS-TuM8, 2 - M --Menyhard, M.: AS-TuM4, 1 - P --Page, S.P.: AS-TuM2, 1 Plano, R.J.: AS-TuM11, 2 Prenitzer, B.I.: AS-TuM9, 2 - R --Reich, D.F.: AS-TuM5, 1 --S-Schuerlein, T.J.: AS-TuM7, **1** Shofner, T.L.: AS-TuM9, 2 Stevie, F.A.: AS-TuM9, 2 Strossman, G.S.: AS-TuM7, 1 Sulyok, A.: AS-TuM4, 1 --T-Tarasevich, B.J.: AS-TuM1, 1 Tielsch, B.J.: AS-TuM2, 1 Turner, N.H.: AS-TuM3, **1** --W-Wang, L.-Q.: AS-TuM1, 1 Wu, K.J.: AS-TuM7, 1