Thursday Morning, November 16, 2006

Applied Surface Science
Room 2005 - Session AS-ThM

Ultra Thin Films and Buried Interfaces

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

8:00am **AS-ThM1 Deep Core X-ray Photoelectron and Auger Spectra: A Comparison of Different Methods for Interpretation**, ***L. Kövér,*** Institute of Nuclear Research of the Hungarian Academy of Sciences, Hungary*; M. Novák,* Institute of Nuclear Research of the Hungarian Academy of Sciences*; S. Egri,* University of Debrecen, Hungary*; I. Cserny, Z. Berényi, J. Tóth,* Institute of Nuclear Research of the Hungarian Academy of Sciences*; W. Drube,* Synchrotron DESY, Germany*; F. Yubero,* CSIC-U, Spain*; S. Tougaard,* University of Southern Denmark*; W.S.M. Werner,* Vienna University of Technology, Austria

The accuracy of quantitative chemical analysis of surface/interface layers is significantly influenced by the spectral contribution from electrons suffered energy losses within the solid. There is little information on the effects of such loss processes for higher energy electrons, promising for analysis of buried interfaces. Deep core (1s, 2s) photoelectron and KLL Auger spectra (resonant and non-resonant) excited by hard X-rays from homogeneous semiconductors Si, Ge and 3d transition metals (Cu, Ni, Fe) were measured with high energy resolution. The measured spectra were analyzed applying different approaches: i) the spectra were fitted by components reflecting surface, bulk and intrinsic (hole induced) excitations, using the "modified Hüfner model",@footnote 1@ ii) by the help of the Partial Intensity Analysis method@footnote 2@ contributions from bulk and surface excitations were successively removed from the spectra, iii) the spectra were simulated using the dielectric response theory.@footnote 3@ For surface excitations, both former models yield only small contributions to the spectra, while using the dielectric response theory surface and bulk excitations are not separated. Strong deviations occur, however, regarding the contribution from intrinsic excitations estimated by using different models, especially between the dielectric response theory and the other two models. Possible reasons of these deviations are discussed. This work was supported by the European Community - Research Infrastructure Action under the FP6 'Structuring the European Research Area' Programme (through the Integrated Infrastructure Initiative) Integrating Activity on Synchrotron and Free Electron Laser Science. @FootnoteText@@footnote 1@L. Kövér, M. Novák, S. Egri, I.Cserny, Z. Berényi, J. Tóth, D. Varga, W. Drube, F. Yubero, S. Tougaard, W. S. M. Werner, Surf. Interface Anal. 38(2006)569. @footnote 2@W. S. M. Werner, Surf. Interface Anal. 31(2001)141. @footnote 3@F. Yubero, S. Tougaard, Phys. Rev. B71(2005)045414.

8:20am **AS-ThM2 Film Thickness Determination of Ultra Thin HfO@sub 2@ Dielectrics with Angle Resolved XPS**, ***W.S.M. Werner,*** *W. Smekal,* Vienna University of Technology, Austria*; D.W. Moon,* Korean Research Institute for Standards and Science, Korea*; K.J. Kim,* Korean Research Institute for Standards and Science, Republic of Korea*; C.J. Powell,* National Institute of Standards and Technology

Reflection electron energy loss spectra (REELS) have been measured for medium energy (300-3400 eV) electrons reflected from solid Si, SiO@sub 2@ and HfO@sub 2@-surfaces. The normalized differential probability for surface and volume excitations was extracted from these data. Furthermore, the total inelastic mean free path (IMFP) for volume scattering as well as the total surface excitation probability (SEP) for a single surface crossing were determined. Measured angle-resolved XPS spectra of thin HfO@sub 2@ films on amorphous Si were analyzed using the experimental electron scattering data. The experimental spectra were compared with model calculations using the SESSA software providing a verification of the commonly employed models for overlayer thickness determination with XPS. Guidelines are given for the optimum experimental conditions and parameters to use in the analysis of angle resolved XPS measurements of ultrathin dielectric films.@footnote 1@ @FootnoteText@ W. Smekal, W. S. M. Werner and C. J. Powell, Surf. Interface Anal. 37(2005)1059@footnote 2@ http://www.nist.gov/srd/nist100.htm

8:40am **AS-ThM3 Structure and Properties of Ultra-Thin SiO@sub 2@ Plasma Polymer Films at Polymer/Metal Interfaces**, ***T. Titz,*** *K. Wapner, G. Grundmeier,* Max-Planck-Institut fuer Eisenforschung, Germany

Thin film surface engineering is one of the key interests of the flat metal producing industry to fabricate ultra-thin functional films in a continuous process. The requirements range from corrosion protection of the surface to a specific control of the physical, optical and electronic properties of the surface. Grundmeier et al. studied in detail the nucleation and growth, wettability and barrier properties of protective plasma polymers on metals.@footnote 1-3@ In the presented work, ultra-thin silica like films have been deposited on zinc and zinc alloy model substrates by plasma polymerization in a microwave plasma reactor. The chemical composition of the films was measured by means of Fourier transform infrared reflection-absorption spectroscopy (FT-IRRAS) as well as X-ray Photoelectron Spectroscopy (XPS). The topography of the plasma polymer films was mapped by atomic force microscopy (AFM). Current density-potential curves for the measurement of the kinetics of the oxygen reduction and electrochemical cyclic voltammetry for the analysis of the redox properties of the plasma modified passive film were performed. Scanning Kelvin Probe studies reveal the electronic properties of the buried interface between the plasma polymer and the metal oxide interface. A clear inhibition of the ion and the electron transfer reactions at the buried interface was found even at very low film thickness. @FootnoteText@ @footnote 1@ G. Grundmeier, P. Thiemann, J. Carpentier, N. Shirtcliffe, M. Stratmann, Thin Solid Films, 446 (2004) 61.@footnote 2@ V. Barranco, J. Carpentier, G. Grundmeier, Electrochim. Acta, 49 (2004) 1999.@footnote 3@ G. Grundmeier, P. Thiemann, J. Carpentier, V. Barranco Surf. Coat. Tech., 174 (2003) 996.

9:00am **AS-ThM4 Interfacial Interactions of PEKK Polymer Coatings onto Oxide-free Phosphate Films on an Aluminum Surface**, *A.L. Asunskis,* ***P.M.A. Sherwood,*** Oklahoma State University

In a series of papers we have shown how thin (10nm or less) oxide free phosphate films can be formed on a number of metals. The films formed have potential as corrosion resistant films. We have also shown that it is possible to extend the range of the surface coatings that can be formed by placing a thin polymer layer over the phosphate layer. In this paper we show how the water insoluble polymer poly(ether ketone ketone), PEKK can be placed over a thin oxide free phosphate film on aluminum metal. The surface and the interfaces involved were studied by valence band and core-level X-ray photoelectron spectroscopy (XPS). Difference spectra in the valence band region were used to show that there is a chemical interaction between the PEKK and phosphate thin films on the aluminum metal. Three different phosphate film compositions were studied using different phosphorous containing acids, H@sub 3@PO@sub 4@, H@sub 3@PO@sub 3@, and H@sub 3@PO@sub 2@. This type of interaction illustrates the potential of phosphates to act as adhesion promoters. The valence band spectra are interpreted by calculations.

9:20am **AS-ThM5 DualBeam and Electron Microscopy Characterization of Buried Interfaces**, ***L.A. Giannuzzi,*** FEI Company **INVITED**

A DualBeam instrument is a focused ion beam (FIB) column and a scanning electron microscope (SEM) on the same platform. The synergistic combination of FIB and SEM enables both 2D and 3D characterization of buried interfaces where site specific precision and throughput may be critical. Interphase interfaces as well as internal interfaces (i.e., grain boundaries) often govern material behavior and properties. Direct DualBeam characterization as well as DualBeam specimen preparation for subsequent transmission electron microscopy (TEM) or scanning TEM (STEM) is often the only way to characterize, monitor, or study the failure analysis mechanisms of buried interfaces, especially where nanometer scale devices or material multi-layers exist. Examples of techniques, methods, and applications of DualBeam characterization and specimen preparation of buried interphase interfaces and grain boundaries will be presented.

10:00am **AS-ThM7 Applications of ToF-SIMS Depth Profiling to Problems involving Buried Interfaces and Particulate**, ***K.G. Lloyd,*** The DuPont Experimental Station

Buried irregular interfaces and particulate present special challenges in terms of chemical analysis and identification, and yet are critically important to diagnose in the manufacture of electronic materials and devices. Cross-sectioning at the right location is often difficult, and, while dual-beam SEM/FIB instruments can often provide excellent visualization of buried defects, matching chemical analysis may be absent or problematic. ToF-SIMS depth profiling, with its ability to acquire spatially-resolved depth profiles while collecting an entire mass spectrum at every "voxel", offers a way to re-visit the problem of buried defects. Unlike traditional dynamic SIMS applications, the emphasis will be on the qualitative, not the quantitative. Multivariate analysis of the overwhelming amount of data can reduce the output from essentially a depth profile at every mass to a small set of chemically-meaningful factors. Examples of

ToF-SIMS depth profiles of relatively homogeneous layers, severely inhomogeneous layers, and buried particulate will be discussed.

10:20am **AS-ThM8 Vacuum Splitting at Buried Electronic Interfaces: A Technique Enabling Nanoscale Chemical and Physical Analysis**, ***W.F. Stickle***, Hewlett-Packard Company; *D. Ohlberg, J.J. Blackstock, C.L. Donley, D.R. Stewart,* Hewlett Packard Labs

In modern solid-state electronic devices, the critical layers and interfaces are now reaching scales of only a few nanometers thick. These layers are generally buried underneath thick and complex materials stacks, the fabrication of which frequently alters the chemical and physical properties of the critical interfaces. Nonetheless, correlating the chemical and physical structure of these nanoscale layers and interfaces is essential for optimizing reliable device performance. The conventional approaches for analysing deeply buried layers employ ion-milling to depth-profile the materials stack down to the critical film or interface. However, such depth-profiling processes often induce chemical and physical changes several nanometers below the exposed surface. This modification of the device stack can reduce the utility of such investigations -- particularly when the critical interfaces are of chemical compositions or physical structures that are easily altered by atom bombardment. We present a novel method for investigating the undamaged physical and chemical properties of buried critical layers and interfaces. This technique is based on engineering a weakened interface adjacent to the layer(s) of interest in the device stack, followed by physical cleaving at this interface in a UHV environment. We present UHV XPS and STM data from a series of experimental nanoelectronic device stacks investigated with this new method, and compare the data against those acquired using conventional depth-profiling. Direct comparison illustrates the utility of this new method for acquiring accurate information on the physical and chemical structure of buried nanoscale layers and interfaces.

10:40am **AS-ThM9 SIMS Measurements of C contamination in SOI**, ***M.H. Yang***, *L. Wang, L. Li,* Evans Analytical Group

SIMS have been shown as a powerful technique to distinguish SOI metal contamination and dopant distributions on surface, in the upper Si layer, in the BOX and at the BOX/Si substrate interface with excellent detection limits.@footnote 1,2@ C measurements in SiO2 has been a challenge for SIMS because in situ carbon absorption in the oxide during SIMS measurements. With improved charge neutralization and special treatment of SOI sample surface including advanced polishing, we are able to achieve C detection in SiO2 at ~1E+17 atoms/cm3 for samples with SOI thickness greater than 10 microns.. We are also able to reduce the surface C tailing effect by an order of magnitude, and achieve better detection of C in the SiO2 BOX and their interfaces for samples with thickness less than 100nm. @FootnoteText@ @footnote 1@Ming Hong Yang, Alice Wang, Monica Neuburger, and R. S. Hockett, "SIMS Measurements of Metal Contamination in SOI," in Silicon-on-Insulator Technology and Devices XII, edited by G. K. Celler, S. Cristoloveanu, F. Gamiz, J. G. Fossum and K. Izumi, Electrochemical Society PV 2005-03, pp. 149-154 (2005). @footnote 2@Stephen P. Smith, Shaw Wang, Ihab Abdelrehim, and R. S. Hockett, "SIMS Measurements of Dopants in SOI Wafers," in Silicon-on-Insulator Technology and Devices XII, edited by G. K. Celler, S. Cristoloveanu, F. Gamiz, J. G. Fossum and K. Izumi, Electrochemical Society PV 2005-03, pp. 363-370 (2005).

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