Monday Morning, November 3, 2003

Applied Surface Science
Room 324/325 - Session AS-MoM
Practical Surface Science
Moderator: P.M.A. Sherwood, Kansas State University

8:20am **AS-MoM1 Aqueous-derived Planar Proxies: A Connection between Surface Science and Real World Catalysts**, *C.F. Conrad,* Virginia Institute of Marine Science*; C.J. Chisholm-Brause,* ***M.J. Kelley,*** College of William & Mary

Real catalysts typically comprise metal or metal oxide nanoclusters on a high-surface area insulator oxide support, prepared by aqueous chemistry. Researchers seeking to overcome the experimental difficulty of studying such materials with surface science techniques have made model catalysts by physical deposition, akin to microfabrication technology. It has now become possible to prepare planar proxies by all-aqueous methods, closely akin to those for real catalysts. After obtaining a hydrous gamma alumina layer on a metal foil, established aqueous solution techniques were used to prepare planar proxy and high surface area materials together. To verify their equivalence, both sets of materials were examined by ToF/SIMS, XPS, SEM/EDS and EXAFS, and by response to organic probe molecules.

8:40am **AS-MoM2 The Role of XPS in Materials Characterization in an Industrial R&D Setting**, ***M.C. Burrell,*** GE Global Research **INVITED**

X-ray photoelectron spectroscopy (XPS) is a widely used method in fundamental surface science and applied materials characterization. As an analytical technique, XPS is an integral part of a modern materials research laboratory. Characterization of complex materials usually requires combinations of analytical methods to provide an understanding of structure-property relationships. In this talk, I will review the types of information provided uniquely by XPS, and illustrate how this information is coupled with data provided from other methods in the characterization of surfaces and thin films. Some examples will be selected from the author's own experience as an XPS expert within a larger materials characterization group at a major industrial R&D site. In this setting, a wide variety of sample types and issues are encountered. The variable degrees of success in the application of XPS to quantitative analysis, determination of oxidation states and functional groups, and thin film compositions will be described. In addition, the current and prospective applications in emerging fields such as biotechnology and nanotechnology will be discussed.

9:20am **AS-MoM4 Quantitative Depth Distribution Analysis of Hg and Na in Glass**, ***T.A. Dang,*** *T.A. Frisk, M.W. Grossman, C.H. Peters,* Osram Sylvania

Fluorescent lamps use mercury for efficient conversion of electrical power to light. During lamp operation, some Hg is consumed through interaction and/or deposition on lamp components. One of the primary sites for interaction is the soda lime glass used as a lamp tube. The association of Hg and Na from the glass has been observed by XPS mapping.@footnote 1@ It is of particular interest to also evaluate the relationship of these two elements in deeper layers. A depth distribution analysis of Hg and Na would readily provide such information. It has always been a challenge to obtain the depth distribution of Na in glass using sputtering techniques. Due to the high mobility of Na, a soft sputtering condition, which minimizes Na diffusion, is generally required. Unfortunately, this also decreases the sensitivity for Hg, whose intensity is several orders of magnitude lower than that of Na. Simultaneous measurement of Na and Hg is strongly desirable because of the non-uniform nature of the interaction. In this presentation, we are going to compare the depth distribution analysis of Na and Hg using Secondary Ion Mass Spectrometry (SIMS) and High-Frequency Square-Wave Sputtered Neutral Mass Spectrometry (HFSW-SNMS). The advantages and disadvantages of each technique will be discussed. Samples included in the evaluation are composed of both quartz and soda lime glass wafers implanted with Na and Hg respectively as well as the real soda lime glass lamp tube subjected to normal lamp operation. @FootnoteText@ @footnote 1@ T. A. Dang, T. A. Frisk and M. G. Grossman, Anal. Bioanal. Chem., 373, 560 (2002).

9:40am **AS-MoM5 Rutherford Backscattering Quantification of Mercury Interaction with Fluorescent Lamp Materials**, ***C.H. Peters,*** *M.W. Grossman, T.A. Dang, T.A. Frisk,* Osram Sylvania Inc.

Understanding mercury interactions with lamp materials is essential to reducing the amount of Hg required for operation of fluorescent lamps as well as improving light output. Rutherford backscattering spectrometry (RBS) is especially well suited to quantification of heavy elements such as Hg or Ba in a light matrix (e.g. soda lime glass). It provides quantitative measurement of trace amounts of mercury and non-destructive depth distribution information. We have developed a method to quantify buried Hg layers in glass under intact 1 to 2 µm thick alumina particulate coatings without the need to remove the coating layer. In uncoated lamps RBS can measure Hg uptake in soda lime glass within the first hour of operation. We have used RBS to characterize the effectiveness of coating layers in reducing interaction between Hg and the glass lamp envelope. Results from actual fluorescent lamps will be compared with quartz and soda lime glass wafers implanted with known amounts of mercury.

10:00am **AS-MoM6 Investigation of the Tribological System of Roller Bearings with TOF-SIMS**, ***U. Gunst,*** *D. Lipinsky,* Westfälische Wilhelms-Universität Münster, Germany*; W.-R. Zabel, G. Poll,* Universität Hannover, Germany*; H.F. Arlinghaus,* Westfälische Wilhelms-Universität Münster, Germany

Tribology is a term describing an important and often complicated set of topics involving friction, lubrication, and wear. The surface characterization of tribological systems is of high importance to enhance their lifetime and to reduce economical loss. We have used time-of-flight secondary ion mass spectrometry (TOF-SIMS) in order to characterize the composition of tribosurfaces and of triboboundaries of high speed rolling element bearings. Different TOF-SIMS methods (static TOF-SIMS, imaging, and depth profiling) were applied to investigate the elemental and molecular surface compositions as a result of interacting surfaces, friction, lubrication, and wear. Using static TOF-SIMS, semi-fluid lubricants, additives, the bearing steel, and the cage material were analyzed to establish reference information. The greases and roller bearing steel surfaces were investigated again after performing tribological test runs with real bearings under almost real application conditions. For these tests the additivation of the lubricants was varied using different primary, ashfree antioxidants. We used TOF-SIMS imaging for the characterization of these tribosurfaces within the race way of angular contact ball bearings. The depth composition of tribological boundary layers was analyzed by performing TOF-SIMS dual beam depth profiling on the top of roller bearing balls, as well as in the bearing race ways. Significant elemental and molecular species were found for lateral and depth distributions of different tribological layer regions - giving correlations to tribological models and interaction mechanisms.

10:20am **AS-MoM7 Interfacial Analysis between Amorphous Carbon Films and Glass by XPS and Improvement of Adhesion Strength at the Interface by Plasma Treatment**, ***S. Takeda,*** *S. Suzuki,* Asahi Glass Co., Ltd, Japan

Amorphous carbon (a-C) films are widely applied in data storage and microelectronic industries because of their unique properties such as excellent hardness, good wear resistance, low friction coefficient, good chemical resistance and high electrical and thermal conductivities etc. However, in case of deposition of the a-C films onto glass, adhesion strength of the films to glass substrate is very poor and the film is easily delaminated at the interface between the film and glass. This is a serious problem for practical applications. Namely, improvement of the adhesion strength is a key issue to apply the a-C films to practical products. In order to overcome this problem, we investigated the effects of plasma treatment in Ar, N2 and O2 prior to the film deposition on the adhesion strength of the films to glass substrate. In the presentation, we report a major factor governing the adhesion strength based on the interfacial analysis between the a-C films and glass by X-ray photoelectron spectroscopy.

10:40am **AS-MoM8 Determination of SiGe Film Composition and Thickness by Combined AES and Multiple-Voltage X-ray Emission Analysis**, ***J.T. Armstrong,*** *S.A. Wight, R.B. Marinenko, D.S. Simons, E.B. Steel,* National Institute of Standards and Technology

SiGe epitaxially grown on Si is used for bandgap-engineered devices with significant potential in a variety of microelectronic products. The composition of SiGe layers is often determined by RBS; however, the analytical accuracy is limited to ~5-10%, in part due to the lack of suitable standards. We are working on development of higher accuracy analytical procedures to characterize SiGe films being considered as NIST reference materials (50-150 nm, 3-40 atom % Ge, grown on Si substrates). We use a combination of Auger electron spectroscopy, multiple voltage x-ray emission analysis, and SIMS to determine their homogeneity, thickness and composition. Results are compared to measurements made on a series of bulk SiGe alloys also being investigated as possible NIST reference materials. The new generation of field emission scanning Auger microprobes (FE-SAM) provides superior capabilities for this type of analysis. With our newly installed FE-SAM, we are able to simultaneously perform Auger electron spectroscopy and high precision and accuracy EDS

x-ray analysis at beam energies ranging from <1 to 25 keV. Using low voltage EDS analysis (2-4 keV) (augmented by the lack of contamination enabled by UHV and incorporating our newly refined correction procedures) we are able to determine the film compositions to better than 2% relative. By multiple higher voltage x-ray analysis (5-25 keV) on the FEASAM and electron microprobe, we are able to determine the lateral homogeneity and thickness. By AES and SIMS we are able to determine the in-depth homogeneity of the films and separately estimate their thickness. Preliminary results show good agreement among these measurements.

11:00am **AS-MoM9 Carbon Gold Sulfide by 13.56 MHz Plasma CVD and Sputtering Process**, ***M.A. Kashem**, S. Morita,* Nagoya University, Japan

Co-operation process of plasma CVD and sputtering is a well-known technique to fabricate metal containing carbonaceous film, however the metal was mixed in the film with polycrystalline structure at content more than a few atomic %.@footnote 1@ CH@sub 4@ and SF@sub 6@ mixture gas plasma induced a unique reaction of HF dissociation and carbon sulfide could be synthesized.@footnote 2@ A new carbon gold sulfide film was formed with using CH@sub 4@, SF@sub 6@ and Ar mixture gas plasma CVD and gold plate discharge electrode. The process was observed and discussed with using a mass spectroscopy. The gold atom was observed to distribute uniformly caused on chemical bond with carbon and sulfur. The carbon gold sulfide structure was confirmed by x-ray diffraction, XPS analysis and refractive index measurement on the effect of thermal treatment at 200 °C. The chemically bonded carbon gold sulfide suggested to be conductive. Therefore, the density of carbon gold sulfide molecular group was observed to affect on a dielectric and conductive property. @FootnoteText@@footnote 1@L. Marutinu, Solar Energy Materials 15, p.21 (1987). @footnote 2@M. Matsushita, Md. Zarid. Bin Harum, Md. Abul Kashem and S. Morita; J. Photoplolym. Sci. and Tech., 12 (1) (1999) pp.11-14.

11:20am **AS-MoM10 Thickness, Dose and Distribution Measurements of Silicon Oxynitride Ultra-thin Films**, ***R.K. Champaneria**, P. Mack, R.G. White, J. Wolstenholme,* Thermo Electron, UK

The continuing requirement for smaller equivalent oxide thickness (EOT) for transistor gate dielectrics has lead to the introduction of new materials with higher dielectric constants than silicon dioxide (high-k dielectrics). At present, silicon oxynitride is an important material for this application. X-ray photoelectron spectroscopy (XPS) is a well-known surface analytical technique. It can provide quantitative information, not only about chemical elements but also their chemical state. The information depth of the technique varies with the material under investigation but is in the region of 5-10 nm for materials commonly encountered in semiconductor device fabrication. This information depth can be controlled by means of the angle at which the photoelectrons are collected (angle resolved XPS or ARXPS) and, by collecting the signal at a number of angles, it is possible to generate a concentration depth profile. Since no material is removed in the generation of the concentration profile, the method is essentially non-destructive. Data will be shown to illustrate how ARXPS can provide accurate and precise measurements of thickness and nitrogen dose in oxynitrides. It will be shown that XPS measurements at a single angle cannot provide accurate measurements of the dose. Using line scans or maps, the uniformity of thickness and dose across a wafer can be measured. ARXPS data can also be used to reconstruct concentration depth profiles. These profiles reveal both the total dose and the distribution of nitrogen in each of its chemical states. It will be shown that methods of profile generation involving sputtering can produce misleading results.

Author Index

Bold page numbers indicate presenter

— A —
Arlinghaus, H.F.: AS-MoM6, 1
Armstrong, J.T.: AS-MoM8, **1**
— B —
Burrell, M.C.: AS-MoM2, **1**
— C —
Champaneria, R.K.: AS-MoM10, **2**
Chisholm-Brause, C.J.: AS-MoM1, 1
Conrad, C.F.: AS-MoM1, 1
— D —
Dang, T.A.: AS-MoM4, **1**; AS-MoM5, 1
— F —
Frisk, T.A.: AS-MoM4, 1; AS-MoM5, 1
— G —
Grossman, M.W.: AS-MoM4, 1; AS-MoM5, 1
Gunst, U.: AS-MoM6, **1**
— K —
Kashem, M.A.: AS-MoM9, **2**
Kelley, M.J.: AS-MoM1, **1**
— L —
Lipinsky, D.: AS-MoM6, 1
— M —
Mack, P.: AS-MoM10, 2
Marinenko, R.B.: AS-MoM8, 1
Morita, S.: AS-MoM9, 2
— P —
Peters, C.H.: AS-MoM4, 1; AS-MoM5, **1**
Poll, G.: AS-MoM6, 1
— S —
Simons, D.S.: AS-MoM8, 1
Steel, E.B.: AS-MoM8, 1
Suzuki, S.: AS-MoM7, 1
— T —
Takeda, S.: AS-MoM7, **1**
— W —
White, R.G.: AS-MoM10, 2
Wight, S.A.: AS-MoM8, 1
Wolstenholme, J.: AS-MoM10, 2
— Z —
Zabel, W.-R.: AS-MoM6, 1