Thursday Morning, October 28, 1999

Applied Surface Science Division
Room 6A - Session AS-ThM

Real World Surface Analysis

Moderator: T. Barr, University of Wisconsin, Millwaukie

9:00am **AS-ThM3 The Real World: Surface Analysis Applications and Innovations in Industry**, ***S.J. Pachuta,*** 3M **INVITED**

The term "real world" surface analysis is often used in opposition to "academic" surface analysis. The distinction is conventionally viewed as being a matter of single crystal studies under ultra high vacuum in the academic case, vs. everything else in the real world case. In reality there is, of course, some overlap between the two, but it is true that industrial analysts must often deal with unknown materials under less than ideal conditions. The following is a far from exhaustive list of the challenges: samples with fingerprints and surface environmental contamination; volatile materials; buried layers, both elemental and organic; complex mixtures such as copolymers and blends with multiple additives; submicron features and defects, possibly on the surface but often buried; insulators; samples too large for most vacuum systems, but which must be preserved and cannot be cut; inhomogeneous materials. More often than not, a multi-instrument approach is needed to address industrial surface analysis problems. The three techniques of x-ray photoelectron spectroscopy (XPS), Auger electron spectroscopy (AES), and secondary ion mass spectrometry (SIMS) comprise the backbone of most industrial surface analysis laboratories. Other techniques like infrared spectroscopy and atomic force microscopy are also being increasingly utilized. An important--and sometimes overlooked--aspect of surface analysis is sample preparation, which often turns out to be the key to solving a problem. This talk will use examples from a diverse industrial laboratory to illustrate the synergism between the various surface analysis methods. Emphasis will be placed on organic materials, since a large part of industrial analysis is concerned with polymers and other organics. New sample preparation methods for XPS and time-of-flight SIMS will be described which extend the capabilities of these techniques.

9:40am **AS-ThM5 Effect of Sputtering Gas on Cleaning Al-Based Intermetallics and the Determination of Surface Compositions based on Auger Analysis**, ***C.J. Jenks,*** *T.E. Bloomer, M.J. Kramer,* Ames Laboratory*; J.W. Burnett,* Iowa State University*; D.W. Delaney, T.A. Lograsso, M.F. Besser, D.J. Sordelet, P.A. Thiel,* Ames Laboratory

Argon is the typical gas of choice for sputtering single crystals in preparation for ultrahigh vacuum studies. However, the use of argon when cleaning Al-based intermetallics leads to preferential etching of Al. This can be a problem because of potential phase changes and the need for a consistent surface composition after annealing. We have examined the extent of this preferential etching as a function of ions/cm@super 2@ for helium, neon, argon, and krypton. The intermetallic substrate has a bulk composition of Al@sub 72.8@Pd@sub 18.6@Mn@sub 8.6@. We find for this material that the steady state Al concentration at the surface is about the same for all the sputtering gases examined. However, the number of ions/cm@super 2@ (which is related to time) depends on the sputtering gas used. Also discussed will be the determination of surface composition of Al-based intermetallics by Auger Electron Spectroscopy. For these materials, in particular, the sensitivity factors will differ greatly between the pure elements and the compound matrix because of changes in electron escape depth, electron backscattering, and atomic density. We find that the use of certain standards can lead to erroneous results.

10:00am **AS-ThM6 Failure Mechanisms of Adhesively Bonded Hot Dipped Galvanised Steel Studied by Small Area XPS**, ***R.G. White,*** VG Scientific, UK*; M.F. Fitzpatrick, J.F. Watts,* University of Surrey, UK

One of the most important requirements of an adhesive joint is the retention of strength for an acceptable time on exposure to a hostile environment. Durability is recognized as one of the most significant problems in the adhesive bonding in industry. In a previous paper, small area XPS established that electrochemistry was responsible for initial bond degradation in a phosphated hot dipped galvanised steel (HDGS) lap joint.@footnote 1@ This paper reports a surface analysis investigation of the failure mechanism of adhesively bonded hot dipped galvanized steel that has been exposed to a hostile environment. The failed lap shear joints show areas of apparent interfacial failure, however, these regions are limited to thin strips at the end of the overlap. These "initiation zones" seem to be a result of environmental exposure and appear to act as initiation sites for crack propagation on mechanical testing, acting as "notch like" features. The study of these areas of the failed surface using small area XPS (15 micron resolution) is reported in this paper, with a view to establishing the role of electrochemical activity at the crevice tip and its role in the subsequent joint failure. Acknowledgement : The authors wish to thank British Steel Strip Products and Welsh Technology Centre for the provision of a Research studentship (MFF) and for permission to publish this paper. @FootnoteText@ @footnote 1@J.F.Watts and M.F.Fitzpatrick Surf. Interf. Anal. in press.

10:20am **AS-ThM7 Novel X-ray Sensor Suite for In Situ Optimization of Thin Film Architectures**, ***L.L. Fehrenbacher,*** *D. Palaith, C. Deaton, J. Ullrich,* Technology Assessment & Transfer, Inc.

Advances in compact, high energy x-ray sources and sensitive detectors are creating new opportunities for the use of x-rays for real time and near real time interrogation of thin film properties. A design approach that combines x-ray diffraction, florescence and reflectivity measurements with a thin film deposition system is described. Phase, composition, surface and interfacial roughness, thickness and density of thin films can now be monitored during a thin film deposition process enabling improved control over process deposition parameters. Details of the equipment and examples of the systems used for rapid development of new multilayer thin film architectures as well as production control are provided.

10:40am **AS-ThM8 A 300mm SAM, with EDX and FIB for Full Wafer Defect and Thin Film Characterization**, ***Y. Uritsky,*** Applied Materials, Inc.*; C.R. Brundle,* Applied materials, Inc.

As design rules shrink and thin film stacks get thinner, the semiconductor equipment manufacturing industry is forced to move to more sophisticated approaches for its particle defect and thin film characterization needs, including surface and thin film analysis. In the past we have occasionally supplemented our full wafer (200 mm) SEM/EDX small particle analysis work by SAM, using small cut up pieces of the wafer. We have now installed the first full 300 mm wafer SAM (Smart 300 from PHI), on which we also have traditional EDX and also FIB. The capabilities of this instrument are briefly described here and examples are given of its use to a) find small defects based on navigation from light scattering files, b) comparatively analyze small particles using Auger, EDX, and FIB sectioning, and c) profile films to examine interfaces. With respect to a) above, since light scattering files are usually quite inaccurate (the predicted coordinates can easily be in error by 100's of µm's), it can be very time-consuming, if not impossible, to re-find very small particles (0.1 µm) with low SEM or Auger contrast. Use of a 300 mm capable dark field optical bench/wafer marker (MicroMark 5000) system to update the particle coordinates with +/- 5 µm) accuracy and/or to create laser-made fiducial marks, can accelerate the subsequent particle re-detection/analysis SAM procedure by a factor of 10. Another particular concern to us, since it is a widely used element in semiconductor processing and is very aggressive, is the relative ability of Auger, EDX, and FIB sectioning Auger and EDX, to reliably detect F as opposed to removing it under the probe beam. This is discussed.

11:00am **AS-ThM9 A Study of the Surface Chemistry and Physical Properties Related to Adhesion of the Polyimide Passivation Layer by XPS, FTIR, and Contact Angle Measurements**, ***T. Jiang,*** Micron Technology Inc.*; C.A, Bradbury,* Micron Technology Inc., US*; M. Canavan,* Micron Technology Inc

Adequate die-to-leadframe adhesion is necessary for lead on chip (LOC) package integrity during and after the manufacturing process. Poor adhesion may result in a variety of defects such as die adhesion failure, marginal wire bond, broken wire, and bent leads ultimately leading to electrical failure. Adhesion between the LOC tape and the polyimide passivation is affected by the surface properties of both materials. Understanding the relationships between these properties is important for the elimination of adhesion failures at die attach and for the continuous improvement of the manufacturing process. To this end, die which fail at attach are analyzed and compared with die which exhibit good adhesion characteristics. This study focuses on the surface chemistry and physical properties of the passivation layer using X-ray photoelectron spectroscopy (XPS), Fourier transform infrared spectroscopy (FTIR), atomic force microscopy (AFM), and surface energy. Molecular concentrations and orientations are investigated and related to adhesion failures at die attach.

11:20am **AS-ThM10 Diffusion of Large Molecules on Metallic Surfaces using TOFSIMS**, ***R. Avci,*** *S.E. Maccagnano,* Montana State University*; G.L. Gresham, G.S. Groenewold,* Idaho National Engineering and Environmental Laboratory

The environmental contamination of clean surfaces creates challenging problems in practical surface analysis. In most cases the contamination is

caused by a contact between a dirty and a clean surface and the subsequent diffusion of the contaminant over the clean surface. In this presentation a semi-quantitative study of the diffusion process of large molecules such as polydimethylsiloxane and tributyl phosphate on metallic surfaces will be presented. Using imaging time-of-flight secondary ion mass spectroscopy (ToFSIMS) we have monitored the diffusion of these molecules by first absorbing them on the surface of a fiber such as linen and nylon and then placing the fiber in contact with a metallic surface such as a gold-coated silicon wafer. ToFSIMS spectra are taken as (a) a function of distance from the contact point and (b) a function of time from moment of contact to determine the diffusion properties of these molecules on the surface. Our preliminary observations show that these molecules rapidly diffuse away from the contact point on the surface of the metal.

11:40am **AS-ThM11 Identification of Surface Chemical Functional Groups in Reverse Osmosis Membranes: An X-ray Photoelectron Spectroscopy Study**, ***S.D. Beverly***, *S. Seal, S.K. Hong,* University of Central Florida

Membrane filtration including reverse osmosis (RO) has emerged as a viable drinking water treatment technology that offers a versatile approach to meeting multiple water quality objectives. Due to fouling and membrane failure, however, wide use of membrane processes for municipal water supplies has not become the reality that it could be. This study is an attempt to identify surface functional groups and chemical changes in surfaces of RO membranes during operation, which would give clues to the nature of the membrane failure. Since the depth of the RO membrane skin layer is less than 50 angstroms, X-ray Photoelectron Spectroscopy (XPS) was chosen to be a practicable analytical tool for this research study. Three commercial RO membranes made of organic polymers of polyamide or cellulose acetate were investigated. These membranes were chosen because of specific characteristics such as chlorine degradation, biological degradation, or fouling resistant coatings. For each membrane, a baseline spectrum was taken and then a sample of the membrane used to treat river water in Tampa, Florida was tested. Each sample was thoroughly rinsed in DI water and allowed to dry before XPS analysis. XPS analysis clearly showed a distinct uptake of chlorine in the polyamide membrane, a probable reason for failure in the drinking water industry. The cellulose acetate membrane showed evidence of amino acids, an indicator of digestion by an unidentified microbe. Based on the findings of this report, future studies are being considered to further investigate chlorine uptake by RO membranes. The studies will include charting chlorine uptake over time and finding limiting factors to chlorine uptake.

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