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

Imaging and Small Area Analysis

Moderator: J.E. Fulghum, Kent State University

8:20am **AS-MoM1 Reading Mechanism Evaluation on Detection of Spontaneous Polarization in a PZT Thin Film**, ***W. Moon,*** Pohang University of Science and Technology, Republic of Korea*; H. Shin, J. Lee, K. Lee, Y.E. Pak,* Samsung Advanced Institute of Technology, Republic of Korea

The methods for detecting a small polarized area in a PZT thin film by SPM techniques are investigated theoretically and experimentally. A small polarized area in a PZT thin film can be constructed and detected by use of SPM. It is found that a 4 μ m by 4 μ m polarized area can be more easily detected by use of EFM techniques under contact-mode-AFM operation. It is shown experimentally as well as theoretically that effects of electrostatic forces are dominant for detection signals for polarized domains in a PZT thin film compared with piezoelectric vibration effects.

8:40am **AS-MoM2 A New Variable Temperature Beam-Deflection AFM**, *A. Feltz,* ***P. Güthner,*** *T. Berghaus,* OMICRON Vakuumphysik GmbH, Germany

AFM experiments at variable temperature are very important for the investigation of phase transitions, growth behaviour, surface diffusion and other temperature depending processes on insulating surfaces with atomic resolution. For this purpose a new Variable Temperature AFM has been developed for minimum drift over a wide temperature range, i.e. AFM operation from 25 K to more than 1000 K. The special optical setup allows to realize a scanned-tip instrument. This allows to limit heating and cooling to the sample realizing a wide temperature range and low drift even for fast temperature changes. The beam deflection technique allows simultaneous topography and lateral force imaging in contact mode, non-contact mode AFM with true atomic resolution, as well as Magnetic Force Microscopy (MFM) and Electrostatic Force Microscopy (EFM). Au(111) was used to study friction at low temperatures. Images of the gold (111) surface in contact mode show the atomic structure and the 23 x @sr@3 surface reconstruction in the topography, and the friction image. At low temperatures down to 30 K the atomic scale friction is drastically increased. True atomic resolution has been achieved in dynamic mode images of silicon (111) 7x7 over a temperature range from 50 K up to about 1000 K. Silicon cantilevers prepared by sputter cleaning of the tip were used for these images. Further experiments will be performed on a non-conducting sample (NaCl).

9:00am **AS-MoM3 AFM Imaging of Thermal Phase Transitions in Polymers**, ***S.N. Magonov,*** Digital Instruments **INVITED**

Atomic force microscopy (AFM) became the leading scanning probe technique, which is used in semiconductor, data storage, and plastics industries. AFM is routinely applied for examination of polymer materials at ambient conditions. Its validity for high-resolution real-space visualization of polymer morphology and nanostructure as well as for compositional mapping of heterogeneous systems had been already recognized. AFM substantially complements other microscopic and diffraction techniques providing visualization of single macromolecules and crystalline polymers, mapping of rubber domains with different cross-linking density and non-destructing imaging of sub-surface features of viscoelastic materials at depths from a few to hundreds of nanometers. Examination of structural changes at thermal transitions is a relatively new AFM capability. Several examples demonstrating such applications will be discussed in this contribution. They include monitoring of structural changes in mesomorphic polysiloxanes, thermal reorganization in block copolymers and visualization of melting and crystallization in ultrathin (20 nm) films of polyolefines.

9:40am **AS-MoM5 Geometry and Tip Effect Simulation in Scanning Kelvin Probe Microscopy**, ***A.E. Efimov,*** Silicon-MDT Ltd., Russia*; S.R. Cohen, I. Visoly, D. Cahen,* Weizmann Institute of Science, Israel

Scanned probe microscopy (SPM) techniques have become an integral part of the fabrication and study of miniaturized devices, as well as of nm-scale features in general. Well-known geometric effects of tip size have been studied and several solutions were proposed for their removal from obtained topographical images. A more subtle effect exists for electronic surface mapping using such modes as scanning Kelvin probe microscopy (SKPM). These effects become significant for high resolution work on surfaces whose topography is not flat. We demonstrate this phenomenon on SKPM studies of CdTe films which exhibit both rich topography and electronic structure at grain boundaries. II-VI semiconductors exhibit interesting electronic properties which not only allow their applications in miniaturized electronic device structures, but suggests their study as model systems as an end in itself. A combined theoretical and computational approach is used to simulate the SKPM imaging process, calculate geometric contributions and separate electronic features from geometric distortions in the scanned probe images, in an attempt to recover the surface potential and therefore move towards true SKPM imaging. The results are compared with parallel techniques, such as scanning tunneling spectroscopy (STS), where the geometric contributions can be effectively ignored.

10:00am **AS-MoM6 Imaging of Particulates using Auger Electrons**, ***M. Prutton,*** *D.K. Wilkinson, M.M. El Gomati, M. Jacka, M. Kirk,* University of York, UK **INVITED**

The currently favoured methodology of scanning Auger microscopy uses UHV SEM systems equipped with field electron emission sources in the column and coaxial mirror or concentric hemispherical electron energy analysers to detect electrons of a selectable kinetic energy. One technological objective is to produce digital maps in which the pixel intensities are proportional to the atomic fractions of the element giving that Auger line in the electron spectrum. This is significantly more difficult than simply acquiring and displaying signals proportional to the height or area of that Auger line because of the dependence of the Auger yield upon the local angle of incidence, the varying composition of sub-surface material and the presence of sharp changes in composition in the vicinity of the current pixel. Most of these difficulties have been removed or minimised in the York Auger microscope (MULSAM). The principal features of MULSAM will be outlined together with a short introduction to the methodology used to combine images obtained from several different scattered electron detectors around the sample. Examples will be given for correction of topographical artefacts in chemical maps and the use of multi-variate statistics to assist in the analysis of particulate catalysts. One disadvantage of current SAM designs is that sequential data acquisition is slow for multi-element samples. A new hyperbolic field analyser (HFA) will be reported that can acquire simultaneously an entire 1024 channel spectrum. It opens the way to obtain entire spectra in each pixel of an image. This could lead to very powerful methods of surface microanalysis using different parts of the spectrum in each pixel to correct for the local topographical and sub-surface composition. Some examples of spectra obtained from particulates using an HFA will be given. The outline design of a spectrum-imaging Auger microscope (SISAM) will be presented.

10:40am **AS-MoM8 Functionalised Plasma Polymer Coatings for Improved Durability of Aluminium-epoxy Adhesive Joints**, ***B.J. Tielsch,*** Kratos Analytical, UK*; M.R. Alexander, G.E. Thompson,* The Corrosion and Protection Centre, UMIST, UK*; T.M. Duc,* BIOPHY Research S A, France*; E. McAlpine,* Alcan International, UK

There is much interest in the use of plasma polymer (PP) coatings to promote adhesion and/or inhibit corrosion of metals.@footnote 1@ These offer environmental and performance benefits over conventional "wet" processing methods such as chromic acid anodising (CAA). Here, we explore the use of carboxylic acid functionalised PP as an adhesion promotion coating on aluminium. The acid functionality, retained from the monomer structure in the plasma deposition process, is intended to form stable chemical bonds with both the aluminium oxide/hydroxide surface and epoxy based adhesives.@footnote 2@ Plasma polymerised acrylic acid and a plasma co-polymer of acrylic acid and octadiene, have been deposited onto plasma etched AA6016 aluminium alloy using RF deposition apparatus. An amine hardened epoxy-based adhesive formulation has been used to bond single lap shear (SLS) joints of PP-coated and CAA alloy. Combining tensile testing of SLS joints with locus-of-failure determination from imaging-XPS and TEM, the relative success of the pretreatments can be gauged. Joint durability was probed by testing joints after storage in water at 60 °C. A decrease in joint strength upon ageing was accompanied by a visual change from failure in the adhesive to failure at a near interfacial region. Chemical state differences in the XPS C1s core level between the acid containing PP and the epoxy containing resin were imaged over the fracture surface. This allowed the effect of PP deposition conditions upon the locus-of-failure and the relationship between this and the failure load to be investigated. Furthermore, information on the distribution of the amine hardener was obtained by imaging of the nitrogen peak. TEM examination of sections taken from the fracture surfaces was correlated with the information obtained using imaging XPS. @FootnoteText@ @footnote 1@R.H. Turner, I. Segall, F.J. Boerio, G.D.

Davis, J. Adhesion 62, p. 1 (1997) @footnote 2@M.R. Alexander, S. Payan and T.M. Duc, SIA 26 p. 961 (1998).

11:00am **AS-MoM9 Glass Transition at Film Surfaces of Polymer Blends with Nanometer Spatial Resolution**, ***F. Dinelli***, University of Washington, US; *C. Buenviaje,* University of Washington; *M. Rafailovich,* State University of New York at Stony Brook; *R.M. Overney, R. Luginbuhl,* University of Washington; *J. Sokolov, X. Hu,* State University of New York at Stony Brook

Surface structure and properties of thin polymeric films are of crucial interest for many technological applications. In ultrathin polymer films we have found that interfacial interactions and local confinement alter the physical properties. Recently we have shown that the glass transition temperature at the surface of thin films can be accurately measured using shear modulation atomic force microscopy (AFM). In this paper we present a first application of this technique to polymer blends (PS/PBMA and PS/PMMA) which, depending on the film thickness and the local spinodal decomposition, offer quite complex folded interfacial regimes. In particular we will present profiles of the glass transition temperature across phase boundaries as a function of film thickness, relative composition and degree of mixing.

11:20am **AS-MoM10 Laser-SNMS and TOF-SIMS Characterization of Sub-µm Structures**, ***R. Kamischke***, *F. Kollmer, A. Schnieders, A. Benninghoven,* Universität Münster, Germany

Sputtering based surface mass spectrometry, as SIMS or Laser-SNMS, combines high sensitivity with high lateral resolution, provided a high fraction of sputtered particles is ionized and a fine focused primary ion beam is applied. Whereas for molecular samples static SIMS is the technique of choice, element analysis can be achieved in addition by dynamic SIMS and by postionization of sputtered neutrals (SNMS). In sub-µm characterization the total amount of material available for an analysis is extremely small. Therefore the transformation probability of a surface atom into an ion should be optimized. The large fraction of sputtered neutrals and their efficient laser-postionization results in a high Laser-SNMS sensitivity. The use of a time-of-flight (TOF) mass spectrometer guarantees parallel mass registration at high transmission. In this contribution we report on recent results of TOF-SIMS and Laser-SNMS characterization of AFM tips (Si@sub 3@N@sub 4@ as well as Si after surface modification or metal loading, respectively) and of sub-µm particles, ranging in size down to 15 nm. The main intention of our investigations was to find out the limits in sensitivity for these two surface mass spectroscopies and to compare both techniques. All experiments were carried out in a gridless reflectron based time-of-flight instrument equipped with a fine focused Ga+ source (< 80 nm) and an excimer laser (248 and 193 nm) for nonresonant postionization. The instrument allows a direct comparison of SIMS and Laser-SNMS results of the same sample. We succeeded in chemical characterization of surface structures (nanotips as well as particles) down to the 10 nm scale by both techniques. Useful yields achieved in SIMS and Laser-SNMS reach 10@super -3@ and 10@super -2@, respectively. As a general result we found that the characterization of the elemental composition of structures with dimensions well below the spot size of the primary ion beam is possible.

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