Applied Surface Science
Room: C-106 - Session AS-ThA

Practical Surface Science II
Moderator: B. Beard, Akzo Nobel Chemicals, Inc.

2:00pm **AS-ThA1 Transferring Classical UHV Techniques into Ambient Pressure - Is the Gap Bridged for Electrons?**, ***A. Vollmer***, University of Cambridge, UK, *J.D. Lipp*, *G.E. Derbyshire*, Rutherford Appleton Laboratories, UK, *H. Weiss*, Universität Magdeburg, Germany, *D. Herein*, ACA, Berlin, Germany, *T. Rayment*, University of Cambridge, UK

Most traditional surface science methods are restricted to studies of model systems under 'ideal' conditions, most prominently single crystals in ultra-high vacuum (UHV). The interest in the development of surface science methods for investigations of systems with a more immediate, practical relevance (e.g., heterogeneous catalysts) remains strong, especially with a view to the difficulties in delineating relationships between interfacial behaviour under conventional "surface science" and "practical" conditions, i.e. high pressure environments. In the field of heterogeneous catalysis the terms "pressure gap" and "material gap" have been coined to describe the relationship between surface science and practical catalysis. Under UHV conditions, gas-surface-interactions are widely studied by means of electron detection (LEED, UPS, XPS, XAS and many more) while in gaseous environments electrons are strongly scattered and quickly attenuated. Until recently,[1] an efficient mode for energy-selective electron detection at ambient pressure was not available. We have now explored the possibilities of gas microstrip detectors (GMSD) as a promising tool for bridging the pressure gap between surface science and 'real world' conditions. We will show that energy-selective electron detection and depth profiling is possible for various systems operating under practical pressure conditions, including powders, layered structures, metals as well as insulators. Investigated systems include partial oxidation catalysts based on Vanadium oxides and phosphates. We will also report upon an ambient pressure surface sensitive scanning X-ray microprobe working under reaction conditions.

[1] T. Rayment et al, Rev. Sci. Instrum. 71 (2000) 3640.

2:20pm **AS-ThA2 Plan to Maximise Information Retrieval from the XPS Survey Scan**, ***J.E. Castle***, The University of Surrey, UK

The work to be described concerns the implementation of procedures designed to extract the maximum information possible from the initial survey scan typically made at the outset of XPS analysis of an unknown surface. The procedures resulted from detailed expert discussion of the issues at the recent 'IUVSTA Workshop No.34'. The procedures devised are intended to give guidance for those developing data systems having a degree of inbuilt inferencing capability. Here we have assessed the recommended procedures using off-line processing where necessary but have, as far as currently possible, mimicked the operation of a hands-off system. We will show in this paper the extent to which information retrieval can be enhanced and discuss the possibilities for this to be implemented in future datasystems.

2:40pm **AS-ThA3 Real World Surface Analysis**, ***W.R. Nieveen***, *T.F. Fister*, *P. Lindley*, Charles Evans & Associates - Evans Analytical Group **INVITED**

In todays short R&D-to-product cycle, there is often an oversimplification of the use and valuation of surface analysis. The dividing line is usually chosen between academic/institutional versus industrial or production environments and the most common divider is typically time. Both arenas are equally "real", but the utilization of equipment and instrumentation is typically quite dissimilar. Consequently, methodology between the two divisions is also substantially different. The outcome of any particular surface analytical experiment may have different significance depending on the environment in which it is conducted. The process by which decisions within the analysis are determined and the resulting decisions from the experiment's conclusion can greatly affect the way a particular analysis is performed. These intangibles frequently affect the perceived success or value of surface analysis. In this talk, we will look at the differences in methodology between typical industrial or production situations compared to the R&D or academic use of surface analysis. We will contrast the deadlines, purpose, and goals of typical R&D projects with the demands, timelines, and expectations of a production problem. We will examine the role of the analyst and the affect his/her experience within and outside the framework of surface analysis has on the results. Current real world examples (from both types of environments) using multiple technique surface analysis will be presented to illustrate the processes. Time permitting, examples of surface analytical methods as a research technique for materials characterization, a method for problem solving and/or failure analysis, and a metrology and/or QA/QC tool will be given. The incorporation of these different environments, the analytical decision processes, and the utilization of the results will be important for "expert system" development in surface analysis.

3:20pm **AS-ThA5 Nanoscale Tomography with the Focused Ion Beam**, ***R. Hull***, University of Virginia **INVITED**

We describe the use of focused ion beam sputtering to create tomographic reconstructions of objects at length scales ranging from tens of nanometers to tens of microns. This is achieved by imaging (with secondary electrons or secondary ion mass spectroscopy, SIMS) the sputtered surface at different depths in the sample. Computer interpolation of successive images then enables reconstruction of the 3D structure and chemistry. The final reconstructions typically contain several million independent voxels of data. We will describe experimental strategies for optimizing the realizable spatial resolution, for example by minimizing generation of topography arising from differential sputtering rates. We will also describe implementation of computer algorithms for interpolating between experimental images. The simplest algorithms employ linear interpolation of pixel intensities between successive images, but these techniques work well only where structures are relatively uniform along the normal to the sputtered surface. For structures with high curvature along the sputtering direction, we employ shape-based interpolation techniques (as developed for the medical tomography field) that enable complex geometrical forms to be reconstructed. Spatial resolution is defined primarily by the probe size and the lateral spread of the incident Ga ions in the sample for lateral resolution, and the implant depth for incident Ga ions and escape depth for secondary electrons/ions for vertical resolution. We have confirmed predicted resolution of order 20-30 nm by direct lateral and vertical detection of 22 nm layers in InAlP/InGaP heterostructures. Chemical sensitivities are greatly limited by the low ionization efficiency of most materials by primary Ga ions. Accordingly, we are exploring photon-based techniques for post-sample ionization of sputtered neutrals. This work is done in collaboration with A. Kubis and G. Shiflet (UVa), D. Dunn (IBM) and D. Backman (GE).

4:00pm **AS-ThA7 Electromigration Behavior of Single Crystal Copper**, *C.M. Contino*, *S.M. Schwarz*, ***L.A. Giannuzzi***, University of Central Florida

A preliminary report on the electromigration behavior of single crystal copper will be presented. A focused ion beam instrument was used to prepare single crystal copper samples oriented along [100] and [110]. Electromigration results of the single crystal samples indicate that the [100] oriented sample had better electromigration properties than the [110] oriented sample.

4:20pm **AS-ThA8 XPS Analysis under External DC and AC Bias**, ***S. Suzer***, *B. Ulgut*, Bilkent University, Turkey

Charging is a nuisance in XPS analysis and is usually circumvented by flooding the sample with low energy electrons. External biasing is an easier/cheaper alternative and can even give additional static or dynamical information about charging which can in turn be related to composition of the sample. In this contribution, we will present XPS spectra of Au/SiO2/Si system under various DC and AC (square-wave) bias conditions and discuss the issues related to charging and/or composition.

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