Introduction to a Special Issue on Surface Analysis

Vincent S. Smentkowski
General Electric Global Research Center, Niskayuna, New York
smentkow@ge.com

The surface, or topmost layers of a material, is the region that is in contact with the environment. The composition and chemistry of the surface often can be drastically different from that of the bulk. For many materials systems (catalysts, coatings, biomedical devices, etc.), the surface chemistry and/or properties determine the device performance. Adhesion, delamination, staining, and corrosion are among the important surface phenomena that need to be understood in industrial settings. Over the past forty years, a number of surface analysis techniques have been commercialized to characterize the composition and microstructure of the surface. The most commonly used techniques are summarized in this issue of *Microscopy Today*.

Symposia at the annual M&M meeting as well as previous articles in both *Microscopy and Microanalysis* and *Microscopy Today* indicate that the microscopy and microanalysis community now considers surface analysis techniques to be important tools that provide data that are complementary to typical microscopy methods such as SEM/EDS. Thus, it seemed like a good time to have an issue of *Microscopy Today* dedicated to surface characterization. This issue contains articles describing Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS—also known as electron spectroscopy for chemical analysis [ESCA]), time-of-flight secondary ion mass spectrometry (ToF-SIMS), low-energy ion scattering (LEIS—also known as ion scattering spectroscopy [ISS]), and an application article showing how XPS provides information that complements that which is obtained by SEM and TEM. Future issues of *Microscopy Today* will have articles on other surface analysis techniques and methods. Scanning probe microscopies (SPM), including atomic force microscopy (AFM), are very valuable surface analysis tools; they are not described in this issue because the November 2010 issue of *Microscopy Today* highlighted these techniques.

As with other microscopy and microanalysis techniques, there is a trade-off as to what the various techniques provide the analyst. The table below shows the major information trade-offs with each technique. In/out refers to the "excitation species" and "detected species," respectively.

Each of the surface analysis techniques described in this special issue can provide spectral information (identify which species are present) and two-dimensional images showing where certain species are present on the surface of the sample. (With ToF-SIMS, one can often image molecular species.) Information about subsurface layers can be obtained by performing depth-profile measurements. Depth-profile measurements employ an erosion cycle between analysis cycles to provide the depth distribution of the species of interest. With ToF-SIMS, for example, one can collect ion images at every depth of a depth-profile measurement and hence can generate 3D images. A full-mass spectrum is saved at every volume element in a ToF-SIMS measurement; thus, ToF-SIMS often reveals unexpected species in sub-surface regions of samples and/or at interfaces. Frequently, these unexpected species turn out to be of importance. AES and XPS have well-defined sensitivity factors facilitating quantitative analysis, however the detection limit of the techniques is less than ToF-SIMS and LEIS. High-resolution XPS spectra are often used to provide oxidation state information about the samples being analyzed. LEIS (ISS) analyses have been performed in a number of research laboratories over the past few decades using either a scanning analyzer or a home-built/custom time-of-flight instrument. The introduction of a commercially available time-of-flight LEIS instrument will surely result in more use of LEIS. LEIS is quantitative and offers the highest surface sensitivity because signals are related to the top monolayer of the surface.

I would like to thank each of the authors who contributed manuscripts to this special issue. I would also like to thank the various researchers who have served as co-chairs for the bi-annual "*Surface Microscopy and Microanalysis in Materials and Biological Systems*" symposia at the 2006, 2008, and 2010 M&M meetings. The idea for this issue grew out of these M&M symposia.

Vincent S. Smentkowski
Vincent S Smentkowski
Guest Editor

Capabilities of State-of-the-Art Surface Analysis Techniques

Technique	In/Out	Lateral Resolution	Sampling Depth	Detection Limit	Elements Analyzed	Molecular Information
AES	Electron/electron	7 nm	2 to 5 nm	~0.1 at%	Z > 2	No
XPS	Photon/electron	3 µm	2 to 5 nm	~0.1 at%	Z > 2	Oxidation state
ToF-SIMS	Ion/ion	60 nm	1 nm	ppm-ppb	All	Direct detection
LEIS	Ion/ion	5 µm	1 atomic layer	10 ppm	*	No

* Can detect elements with a mass greater than the primary ion beam

doi: 10.1017/S1551929511000010