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Utilizing Correlative, Multi-Technique Surface Analysis to Complement Microscopy Studies

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Full chemical characterization of a sample necessitates measurement of both bulk and surface properties. To achieve this, an array of experimental techniques may be employed; allowing the field of view to be narrowed both in the X-Y plane, to investigate changes in lateral surface morphology, and in Z to enable the understanding of varying surface and interfacial chemistry with depth. If it is easy to correlate the data, then building a comprehensive picture of the nature of the sample is possible.

To this end we have used a new, multi-technique surface analysis platform that incorporates micro-focusing X-ray photoelectron spectroscopy (XPS), ion scattering spectroscopy (ISS), reflected electron energy loss spectroscopy (REELS), UV photoelectron spectroscopy (UPS) and Raman spectroscopy to complement imaging analysis completed using EDS and EBSD. The ability to combine high spatial resolution imaging in the X-Y plane, with highly surface sensitive analysis on the new platform.

An example of this approach is shown in figure 1, where a CIGS solar cell device is analyzed using EDS and XPS. The same areas on the surface were analyzed. Areas 1 and 2 are intact areas of the active material, area 3 is an area that has the electrical contact, and area 4 is an area that has delaminated. Compositional analysis shows that the surface measured by XPS, and comprising the outer 10 nm of the sample, is markedly different from the composition determined using EDS. The presence of an indium tin oxide overcoat is shown by XPS, which is too thick to be easily detected via EDS. Comparison of cross-sectional images from the SEM with depth profile analysis from the same device allows accurate determination of the thickness of layers, the compositional variation with the layers (particularly important for the CIGS layer), and establishing exactly where the delamination occurs.

In this presentation it will be shown how this approach is beneficial using additional examples from a range of sample types, including carbon nanomaterials, microelectronics samples, and the analysis of artworks.

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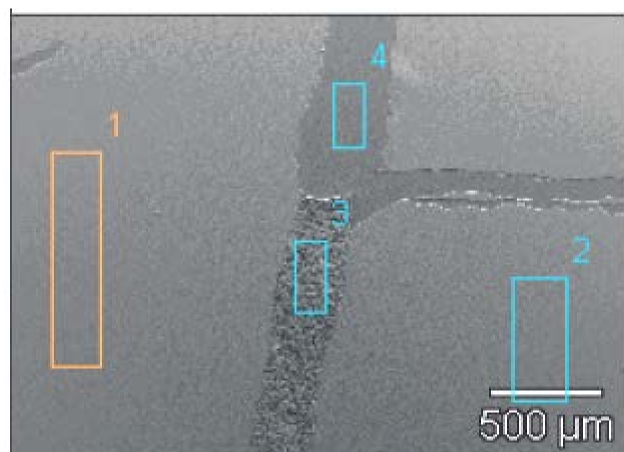

Figure 1. SEM image of the CIGS solar cell sample showing the 4 areas analyzed.

EDS	Ga-K	Zn-K	O-K	Cr-K	In-L	Cd-L	Ag-L	Mo-L	Se-K	Fe-K	Cu-K
Area 1	2.9	2.5	17.4	0.8	24.3	7.4		2.3	22.9	2.4	12.4
Area 2	3.0	2.9	17.6	0.7	25.0	7.8		2.1	21.6	2.5	12.3
Area 3							100.0				
Area 4				17.7				61.0	2.9	18.4	

Table 1. Measured composition by EDS for the 4 indicated areas.

XPS	Ga2p3	Zn2p3	O1s	Sn3d5	In3d	Cd3d5	Ag3d	Mo3d	Se3d
Area 1	0.3	2.5	68.2	2.7	26.0	0.2	0.00	0.2	0.0
Area 2	0.0	2.5	66.6	2.9	27.4	0.2	0.1	0.1	0.2
Area 3	0.0	1.8	65.2	2.6	24.3	0.1	5.5	0.5	0.1
Area 4	0.1	1.4	41.1	1.2	11.6	0.0	0.0	17.1	26.7

Table 2. Measured composition by XPS for the 4 indicated areas.

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