

Making Light Work: Enhancing Surface and Thin Film Analysis through In-Situ Complementary Spectroscopies

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Advanced materials present ever increasing challenges to the analytical scientist. Composite materials built from nanostructures or ultra-thin films, often with complex chemistries present, are now required in a broad range of applications, and achieving full characterization is rarely managed using only one analysis method. To maintain confidence in the results from the utilization of several different methods, it is advantageous to be able to perform experiments on the same platform. Ideally, this should be without having to move the sample, removing the need for additional registration or processing to ensure that the data is being collected from the same position.

For surface analysis, it has been common for many years to incorporate related analysis techniques onto the same instrument. For example, X-ray photoelectron spectroscopy (XPS) systems are commonly equipped with UV light sources to facilitate investigation of additional properties of materials via ultra-violet photoelectron spectroscopy (UPS). The ion source that is typically used for sample cleaning and depth profiling can also be used for low energy ion scattering (LEIS or ISS), providing more surface sensitive elemental composition information than can be delivered from XPS alone. The addition of a focused electron source enables Auger electron spectroscopy (AES) which provides surface sensitive composition at higher spatial resolution than XPS can offer. With the exception of ISS, all these routine additional analysis techniques are electron spectroscopy based, and offer similar information.

We have recently integrated further instrumentation onto two XPS systems. By adding a Thermo Scientific UltraDry detector to a Thermo Scientific ESCALAB Xi instrument, energy dispersive X-ray spectroscopy (EDS) can be performed using the field emission electron source usually used for AES. This enables high resolution elemental information to be collected via EDS, and complimentary XPS data to be obtained from the same position on the sample as part of the same experiment as shown in Figure 1. The X-ray detector can also be used with the monochromated (Al K α or Ag K α), micro-focused X-ray source to collect X-ray fluorescence data, adding further information content. The greater depth of analysis of EDS compared to XPS, allows correlation of surface and bulk composition, without having to damage the surface.

The second integrated system has combined a Raman spectrometer with a micro-focused, monochromated XPS system. The focal points are aligned such that data can be acquired from the same point simultaneously, and that the sizes of the analysis areas are comparable in size. This combined approach is particularly powerful when analyzing carbon nanomaterials, as shown in Figure 2. Chemical modifications of the material can be easily determined and quantified with XPS, and Raman offers a fast way of determining the quality and conformity of the material. The greater depth of field of the Raman spectrometer also offers bulk information to complement the surface sensitive XPS data.

In this presentation we will discuss the strengths of this combined, in-situ approach to surface analysis, illustrated with examples from a range of applications including carbon nanomaterials, microelectronics and geology.

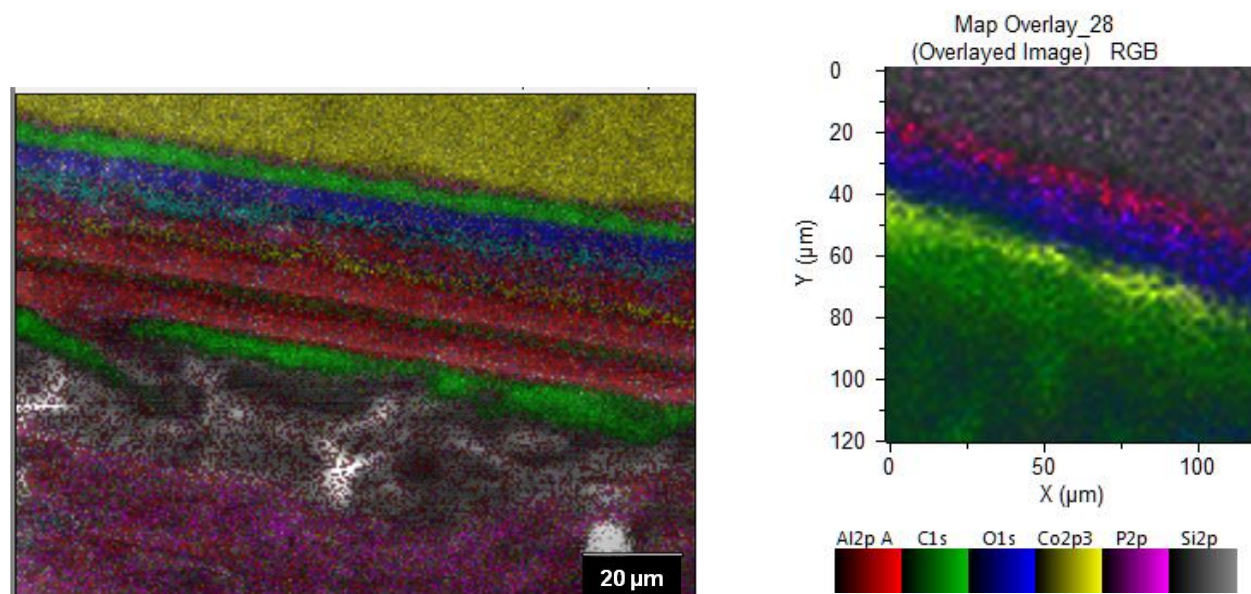


Figure 1. EDS component image and XPS image of the layers in a battery assembly following in-situ ion cleaning. More extensive structure is apparent from the EDS image, whereas chemical state information can be determined from the XPS data.

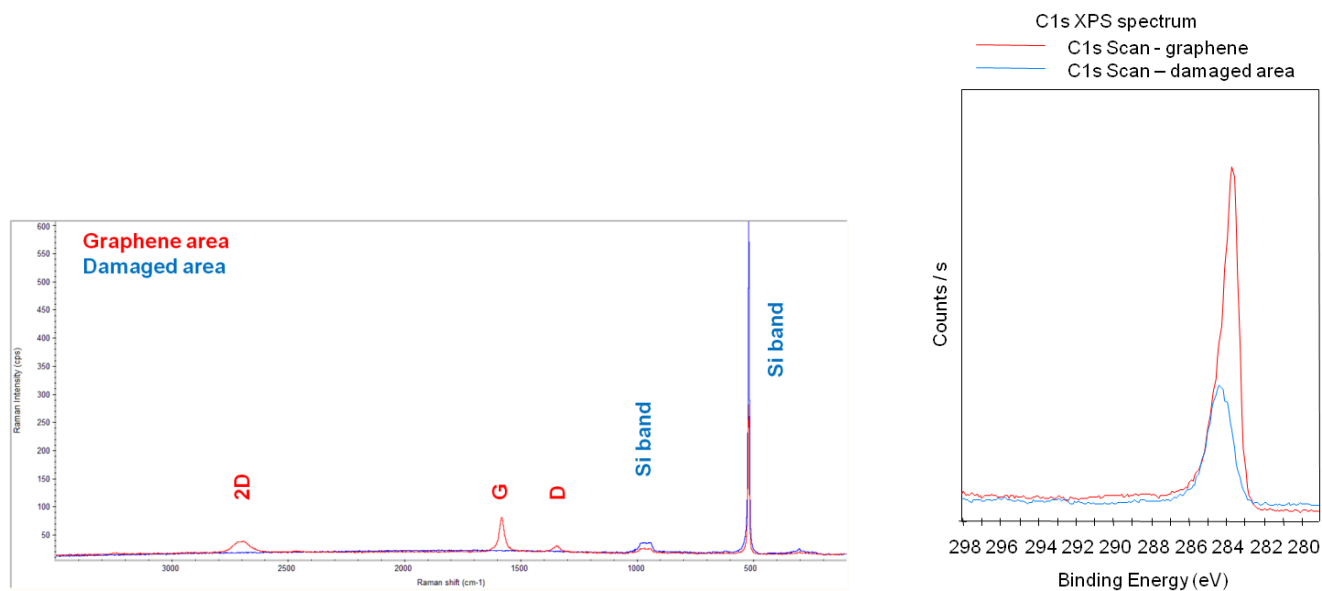


Figure 2. Raman and C1s XPS data from two points on a graphene layer on Si. The red spectra are from the intact graphene layer, the blue spectra from damaged areas on the film.