Journal of Surface Analysis Vol. 26, No.2 (2019) pp. 182-183

Extended Abstract of PSA-19
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Enhancing Surface and Thin Film Analysis Through In-Situ Complementary Raman Spectroscopy

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(Received: May 29, 2019; Accepted: July 20, 2019)

Full characterization of complex materials with ultra-thin film structures or nanostructures can rarely be accomplished with a single analysis technique. X-ray Photoelectron Spectroscopy (XPS) has often been complemented with Ultra-violet Photoelectron Spectroscopy (UPS), ion scattering data or electron microscopy. This presentation will show how the addition of in-situ Raman spectroscopy to an XPS surface analysis system can help to characterize 2D nano-materials such as graphene, by adding chemical and bulk information to surface information all obtained from the same analysis position on the sample.

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 a standard XPS system. The 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. Chemical modifications of the material can be easily determined and quantified with XPS. Raman offers a fast way of determining the quality and conformity of the material, and direct compound identification. The greater depth of field of the Raman spectrometer also offers bulk information to complement the surface sensitive XPS data.

One such example of combined XPS and Raman analysis is that of boron nitride (BN) deposited onto a copper substrate. Boron nitride takes in 2 forms, cubic and hexagonal, and it is the latter form (h-BN) which is of interest as a 2D material, exhibiting high thermal

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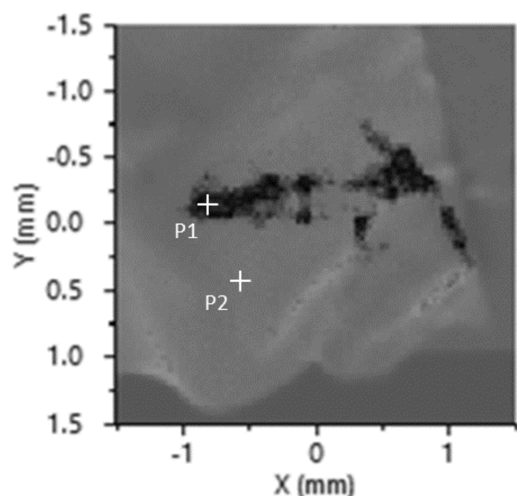

Fig. 1 N1s phase map, showing presence of nitride (dark areas).

conductivity and allowing tuning of electrical properties by functionalization or doping[1,2]. Rapid XPS mapping of the N1s signal demonstrates two states of nitrogen present on the substrate surface, and these two "phases" can then be plotted out to reveal a distribution of on the surface (Fig. 1). XPS point analysis at representative sites P1 and P2 show that boron is present at point P1, suggesting the presence of boron nitride, while point P2 shows no detectable boron signal. While the XPS narrow scan data show some oxidation states in both the N1s and B1s, Raman data collected

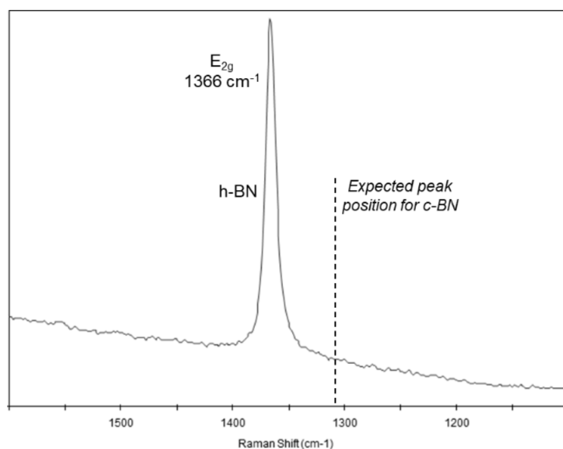

Fig. 2 Raman spectrum from point P1.

from point P1 (Fig. 2) shows a clear h-BN signal, demonstrating that the primary hexagonal structure has been unaffected by any oxidation during deposition onto the copper substrate.

References

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