Characterization of Thin Films and Materials

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Good Practices for XPS Peak Fitting, II

X-ray photoelectron spectroscopy (XPS) has become an extremely important surface characterization tool. It is receiving in excess of 10,000 mentions per year in the literature.[1, 2] This increasing popularity is no doubt due to its many strengths. For example, XPS is quantitative, surface sensitive, able to detect all the elements except hydrogen and helium, sensitive to the oxidation states of the elements, and able to analyze many types of samples. However, it is not the only game in town. Many surface analytical problems are best solved using a combination of different surface and material analytical techniques. For example, XPS and time-of-flight secondary ion mass spectrometry (ToF-SIMS) are often used together, and low energy ion scattering (LEIS) is increasingly being employed with them, where each method enjoys its own unique and complementary strengths.[3-5] Indeed, ToF-SIMS often has much better detection limits than XPS, e.g., for the metals and halogens, and it generally deals better with very complex samples, e.g., biological materials.[4] And, of course, LEIS is more surface sensitive that either XPS or ToF-SIMS, which gives it powerful advantages.[6] Nevertheless, the number of citations XPS is receiving compared to the other techniques makes it clear that it is currently the most important technique for chemically analyzing surfaces, and not just *primus inter pares*.

There are two basic types of X-ray photoelectron spectroscopy (XPS) scans – survey and narrow. Survey scans are taken over a wide energy range – often over 1000 eV or more. In contrast, narrow scans are obtained over a much narrower range – usually over about 20 eV. Both types of scans are regularly discussed and shown in the scientific literature. **Figure 1** shows a simple survey scan and a complex narrow scan. In a survey scan one can often quickly identify the main elements at the surface of one's material (**Figure 1a** shows a material that contains primarily carbon and oxygen). In addition, in survey scans one typically sees rising baselines on the higher binding energy sides of peaks, e.g., around the C 1s signal in **Figure 1a**, Auger signals (the small unmarked signal around 1000 eV in **Figure 1a** is the oxygen Auger signal), signals from the valence band (they come around 25 eV and are hard to see in **Figure 1a**), plasmon loss peaks due to excitation of plasmons by exiting electrons, and multiple peaks from the same element, which act to confirm the presence of a given element, e.g., if we had sulfur at our surface we would expect both the S 2p and S 2s signals. XPS would probably still be an important technique even if it only provided this type of information. However, narrow scans are even richer in information. Narrow scans allow one to home in on a particular element to determine its oxidation state, and whether the element appears in different oxidation states. Narrow scans are the focus of most XPS data work up.[7]

The increasing recognition of the importance of XPS and the increasing number of instruments in the world has drawn some users to the technique that are perhaps not well trained in analyzing its data. For example, as an editor of Applied Surface Science, MRL regularly sees manuscripts with rather poor narrow scan peak fitting in them. Thus, we believe it is important to describe some of the challenges and pitfalls of XPS data analysis, along with ways to circumvent them. Some of our recent journal articles have addressed these issues.[8-10] In addition, our December 2015 ***VT&C*** article reviewed various good practices for peak fitting XPS narrow scans.[11] These included:

(i) Using chi-squared
(ii) Using the Abbe criterion
(iii) Showing the sum of fit components