

Episode 10: XPS, UPS, ISS – Surface Spectroscopic Techniques for Chemical Analysis

Presented By:

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01

Q: When will your in-house XPS testing become available?

A: Available as of 6/11/20

02

Q: Is the intensity peak Lorentzian, Gaussian, or a mix. If Gaussian, or partially so, what is the phenomena responsible for the distribution in binding energies?

A: It's a combination. The contribution of the spectrometer is Gaussian in shape while the natural core level lineshape is Lorentzian. The atomic-level should also be considered, i.e., with metallic species, the ejected photoelectrons may interact with the conduction electrons, losing very small portions of energy which account for the peaks being asymmetric.

03

Q: What is the collection angle of the electron lens for the Nexsa? Is it the same as K-alpha?

A: 60°. Yes.

04

Q: What is the spot size of UPS and ISS?

A: Few hundred microns to a few mm.

05

Q: For the ISS are you limited to the Ar from the ion gun or can other inert gasses be used?

A: Other inert gases can be used like Ne, Ar and Kr. Alkali ion beams may also be used.

06

Q: How to post process the data obtained from xps, outside the xps software

A: I use Origin and Excel outside of the XPS software (CASA, Advantage, Multipak, etc).

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Q: For ISS, what would be the error quantitatively determining the composition of a film composed of heavy atoms (e.g. atomic mass >Si)? What is the penetration depth?

A: The mass resolution and/or sensitivity is insufficient for heavier elements when using He⁺ ions. Additionally, large background from tails are expected for samples with heavier elements resulting in the poorer detection limits. The penetration depth depends on the ion beam energy but usually less than 10 nm with ISS. That is why you go to higher mass for better sensitivity.

08

Q: Why is CaFx not CaF₂?

A: They are the same but "x" is used in case of the presence of nonstoichiometric fluorides on the surface.

09

Q: Is surface roughness a problem for ISS?

A: It could be. The roughness on the surface can sometimes contribute to some of the surface atoms that cannot be seen by the analyzer due to shadowing effects.

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Q: Can depth profiling be done to remove the top layer and also see how composition changes with depth?

A: Yes. This is the point of depth profiling; determining the composition as a function of depth.

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Q: Do powder samples need special preparations before analysis?

A: Powders are usually mounted by pressing it on carbon tape or indium foil. Some make a pellet out of the powder to be able to analyze in XPS.

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Q: What is the uncertainty in the atom% conc of Li?

A: In general, the uncertainty in atomic % is ~ +/- 10%. Weaker signals have much higher uncertainties.

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Q: For the li ion battery expt, why did the Mn conc is higher in the cycled compared to the pristine sample? Where is the extra Mn component coming from? from the bulk?

A: The Mn content on the cycled sample is only slightly higher than that of the pristine sample and it is within the % error.

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Q: For the Li example, do you expect sample nonhomogeneity to affect the composition measurement? What was the analysis area size?

A: Yes. The spot size varies from tens of microns to a few hundred microns or even mm size depending on the XPS instrument.

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Q: Where can I find a collection of Work Function values?

A: I found some here:

<https://public.wsu.edu/~pchemlab/documents/Work-functionvalues.pdf>

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Q: Can ISS measure Hydrogen?

A: Hydrogen has been detected through ISS using a 1 keV 4He⁺ ion beam and narrow scattering angles from 0° to 15° (F. Shoji et. al. Surf. Sci. Lett., 220, 1989, L719-L725). The Hydrogen Forward Scattering Spectrometry is (HFSS) usually used for the detection and quantification of H in films. This is similar to ISS technique but instead of the reflected ion beam, the H atoms that are sputtered away are analyzed using a solid state detector.

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Q: How does charging of the sample affect the XPS signal and how do you mitigate it?

A: As photoelectrons are emitted from the surface, a positive charge will build up on the sample resulting in a shift to higher binding energies of the observed peaks in the spectrum. The resulting spectra may also be broadened significantly. The most common solution to this problem is the use of low energy electron flood gun to overcompensate for the positive charge.

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Q: How well does UPS work for thin films?

A: The information depth for UPS is about 2-3 nm. The usual problem encountered in thin film analysis using UPS is surface contamination. UPS is very surface sensitive that the mere presence of adventitious C due to atmosphere exposure may affect UPS measurements (i.e. valence band spectra, work function measurements). Argon cluster sputtering is usually done prior to the analysis to remove any contaminants without changing the chemistry of the surface.

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Q: I know that an XPS survey scan can contain Auger peaks. Do you ever use this for analysis?

A: Yes. The Auger peaks can sometimes reveal differences in binding energies between oxidation states of some metals and its corresponding XPS cannot (i.e. Cu0 and Cu+1 peaks overlaps in Cu 2p while bigger chemical shifts is observed for these two states in Cu LMM).

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Q: During depth profiling one of the problems with older tools was that the bottom of the crater became rougher and rougher which affected the data. How does roughness affect XPS data?

A: For the depth profile, the sputtering process can cause roughness to occur and this degrades the depth resolution as well as the accuracy of the quantification. This effect is usually mitigated by rotating the sample (azimuthal rotation) during the sputtering part of a profiling cycle.

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Q: I assume if one does an XPS measurements on a porous structure that you get data from both the surface of the material and potentially from inside the pores. Is this correct and how would you verify it.

A: It really depends on the size, geometry and porosity of the material, but it is possible to get the XPS information both from the surface and inside the pores. To confirm, changing the take-off angle to be more surface sensitive will change the relative composition and adding TEM measurements to get a general idea on the differences between the surface and inside the pores.

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