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X-ray Photoelectron Spectromicroscopy

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X-ray Photoelectron Spectroscopy (XPS) has evolved into a powerful analytical technique for the identification and quantification of elements within the first 10nm of a sample surface. Not only is the technique capable of identifying elements with the equivalent of ppb sensitivities, but it can also identify the chemical state or environment of that element. This capability allows the technique to provide chemical information within the surface region, an area of a sample that can determine the functionality and usability of the material in real-world applications.

In the typical XPS experiment, low energy X-rays (Mg or Al Kα) are irradiated on to the sample surface and the subsequent emitted electrons are then energy analyzed revealing not only which elements are present but subtle shifts in their absolute energy enable a determination of the chemical environment or oxidation state to be made. By measuring the number of emitted electrons, as determined by the peak area gives a concentration measurement enabling quantitative results to be provided. Historical measurements focused on large area analysis, but improvements in X-ray source technology and electron energy analyzers opened up small area and imaging capabilities. The recent integration of delay line detector technology enabled quantitative imaging to become a reality. With that the development of the Photoelectron Spectromicroscopy capability has subsequently evolved.

Figure 1 shows a schematic arrangement for the Kratos AXIS UltraDLD imaging XPS instrument including the delay line detection system that provides the quantitative imaging facility. The spectrometer integrates the unique spherical mirror analyzer for imaging that enables a high spatial resolution image to be achieved with high chemical specificity. Figure 2 shows an example of the quantitative images that can be achieved with arrangement. In order to achieve the spectromicrocopy capability the instrument is operated slightly differently from the standard imaging mode. In this case a series of images are obtained at a range of electron energies, (Figure 3). Using imaging stacking methods, spectroscopic data can be extracted from a group of pixels to provide both elemental and chemical information. The use of single or small groups of pixels however leads to poor S/N and a low confidence in the chemical interpretation. By applying Principle Component Analysis (PCA), substantial improvements in the achieved S/N can be obtained even from small groups of pixels that result in a substantially improved ability to identify chemical information. Curve fitting and retrospective application of those components enables the distribution of unique chemical species to be determined.

A benefit of modern XPS instruments is not only in their ability to achieve high spatial resolution images and from that the microscopic analysis capability, but when this capability is combined with stage translation, high spatial resolution images with chemical specificity can be developed over relatively large areas (mm rather than μm). Therefore in conclusion X-ray Photoelectron Spectromicroscpy provides both elemental and chemical information on the micron scale and when combined with stitched imaging techniques can yield valuable information on the surface chemistry of materials over large areas.

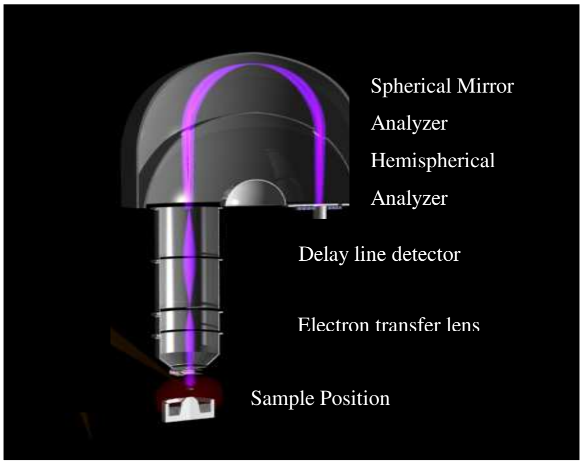

Fig. 1 Schematic arrangement of AXIS UltraDLD Spherical Mirror Analyzer

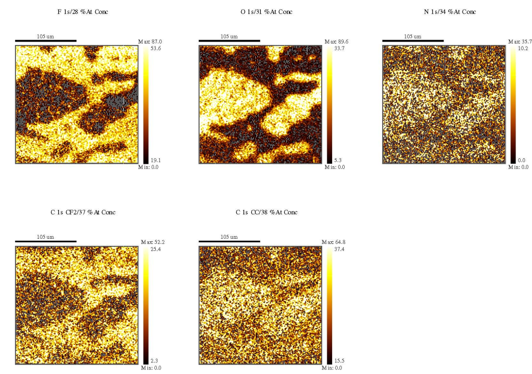

Fig. 2 Quantitative images using the Spherical Mirror analyzer

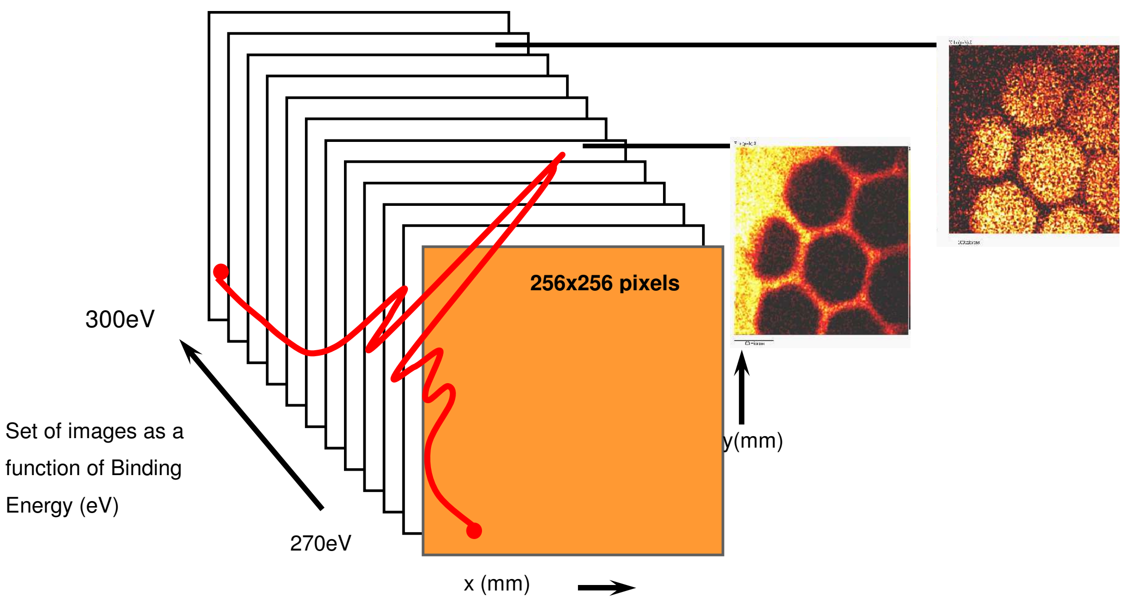

Fig. 3 Derivation of spectra from images (spectromicroscopy)

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