

Quantitative X-ray Photoelectron Spectroscopy Imaging for Small Feature Compositional Screening

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X-ray photoelectron spectroscopy (XPS) is an established technique for *surface* characterization where information originates from the upper 10 nm of the sample surface. XPS has the further advantage of being a quantitative technique. Modern state-of-the-art spectrometers are capable of data acquisition from small, selected areas of the specimen where the analysis area is defined by either using a focused x-ray excitation source or collecting photoelectrons from a selected area defined using an aperture/iris in the electrostatic lens column, sometimes termed a virtual probe. Using either of these approaches to define a small analysis area limits the smallest possible analysis area to *ca.* 10 μm using ISO definition of the 80% / 20% decrease in the signal [1]. However, although approximately 90% of the signal will arise from within the selected area, Baer et al. [2] demonstrated that information is collected from up to three times the defined area which could compromise interpretation of data.

As an alternative to selected area XPS outlined above this paper presents the implementation and application of quantitative XPS imaging which has become possible with the recent implementation of pulse counting 2D detectors for XPS imaging. There are a number of advantages to using quantitative imaging over the more conventional scanned spectrum. When acquiring spectra it must be assumed that the area sampled is homogeneous which in reality may or may not be the case. By acquiring quantitative images either elemental or chemical distribution of the species at the surface can be observed with a spatial resolution in the order of 3 μm . Furthermore, combining fast imaging with stage movements allows the acquisition of high resolution XPS images over areas of several mm^2 .

The spherical mirror analyzer (SMA) incorporated into a standard laboratory spectrometer allows fast parallel images to be acquired whereby the spatial resolution of the photoelectrons at the surface is preserved as they are transmitted through the analyzer. Importantly the SMA works in fixed analyzer transmission mode, such that the energy resolution remains constant as a function of binding (or kinetic) energy. This becomes very important when requiring to quantify images and is analogous to the operation of the hemispherical analyzer used in normal spectroscopy mode.

The familiar photoelectron spectrum comprises a series of peaks corresponding to the elements at the surface of the sample. These peaks sit on a background of inelastically scattered photoelectrons which generally increases with increasing binding energy. The relative atomic concentration of the elements at the surface is calculated using the area under the peak after subtracting the background, and correcting for a relative sensitivity factor and instrument transmission. To generate a quantitative image, the same steps must be undertaken whereby each image acquired for a specific element must have a background image subtracted and be corrected for the relative sensitive factor and instrument transmission. In generating an image that better describes the background for a specific photoelectron peak, images are acquired either side of the photoemission peak itself and a linearly interpolated image calculated which is then subtracted from the peak image. This is shown

in both imaging mode and the equivalent spectroscopic mode in figure 1. A test sample was produced where bulk PTFE polymer was covered with a mask and the surface was modified by exposure to a propanal plasma creating a patterned sample with hydrocarbon islands on a fluorocarbon substrate. Using the imaging technique outlined above quantitative images were generated for hydrocarbon, fluorocarbon, oxygen and fluorine, shown in figure 2. Quantification of the elemental composition was found to be dependent on acquisition time of the images but for a total acquisition time for all images of 405 s the quantification was in good agreement with spectra acquired from a 55 μm diameter analysis area. The advantage of the imaging approach to surface composition determination is apparent from the images where it can be seen that the coverage of the area exposed to the propanal is not homogeneous. Post acquisition the software [3] is used to define small areas within the plasma treated area to generate the relative atomic concentration.

Quantitative imaging has been demonstrated to be a useful way to characterize the surface chemistry of samples. This approach allows the quantification from areas smaller than possible with traditional selected area spectroscopy whilst also identifying areas of inhomogeneity which might otherwise be overlooked in spectroscopy mode.

References:

- [1] ISO 18115:2001
- [2] D.R. Baer, M.H. Engelhard, *Surf. Int. Anal.*, 29, 766-772 (2000).
- [3] CasaXPS processing software, CasaXPS Ltd, UK www.casaxps.com

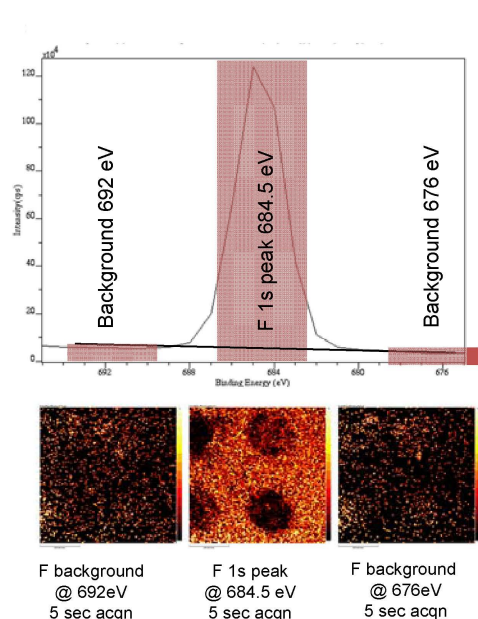


Fig. 1. Example of peak and background images and equivalent for spectra for patterned sample.

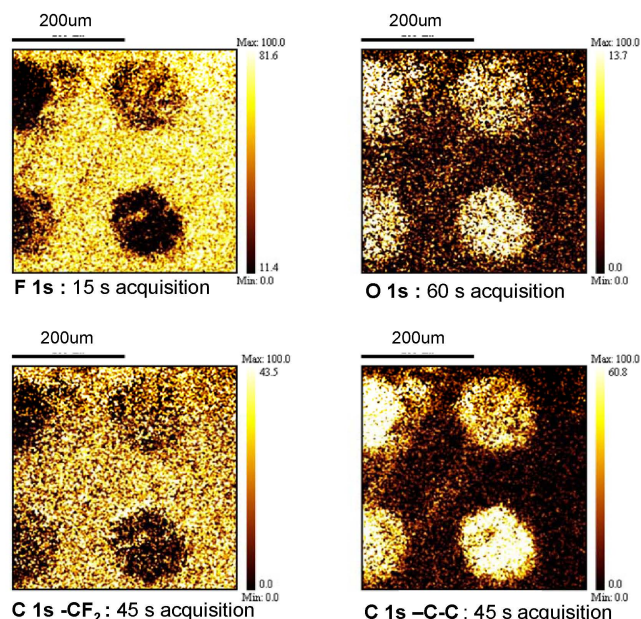


Fig. 2. Quantitative elemental and chemical state images from propanal plasma polymer patterned sample on PTFE substrate.