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Advances in surface chemical state imaging and depth profiling using XPS

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As the critical dimensions of fabricated devices shrink and the explotation of novel nanotechnology effects, each material's surface properties begin to dominate, rather than the bulk properties. These dimensions are such that essentially the entire structure becomes composed of only surface material. Mechanisms to explore these surface properties become paramount to their understanding and characterization for technological development.

Elemental and chemical analysis using traditional x-ray, optical and electron techniques often does not differentiate subtle surface phenomena from bulk material as these techniques generally sample much deeper than the dimensions of many nanotechnology effects. In x-ray photoelectron spectroscopy (XPS), all of the signal comes from within 5-10 nm of the surface and this technique is sensitive to sub-monolayer differences in surface composition and even chemical state, wherein lies its power. XPS provides non-destructive chemical state analysis from the surface of insulating samples and is fundamentally directly quantitative, suffering no substantial matrix effects. This surface chemical signal can be obtained and processed in various ways to generate representative maps or images of the chemistry on a sample surface highlighting any sample inhomogeneities.

Although a counting technique generally beholden to Poisson statistics, similar to XRF, XPS has a distinct background effect, which can completely mask large differences in adjacent regions, if not properly addressed. Figure 1 shows how spectra from two adjacent regions of radically different composition would have nearly constant measured intensity, if no background correction were performed. Improved collection efficiency has limited sample damage, such that imaging of organic, polymeric and biological samples are done regularly, extending to recent work showing that XPS imaging characterization of DNA microarrays can be used as a complement to other surface analytical techniques in the exploration of surface chemistry effects [1].

Surface segregation of bulk materials, or enhancement of surface composition by external treatment is not easily addressed with bulk techniques at the heart of many industrial processes. A particular example exploring combinatorial screening of micro patterned co-polymer arrays for cell adhesion shows how imaging the sample array allows the mixtures with the suitable surface chemistry composition range to be quickly identified [2].

Angle resolved XPS (ARXPS) depth profiling might be an excellent technique for chemical state and molecular orientation determination. To realize these goals the high resolution spectrometer is required with proper and highly efficient charge neutralization and finally with the advanced software for background calculation. The developed advanced Maximum Entropy Method (MEM) software for ARXPS measurements may used to probe the chemical composition of the sample over the first few nm of the sample surface (Figure 2).

Furthermore, the hypothesised close packed surface arrangement with the molecular chains arranged perpendicular to the Si surface is confirmed.

Unlike electron excited techniques, which require conductive surfaces to avoid instantaneous deflection of the primary beam due to surface charging, XPS imaging of insulating surfaces is possible with modern charge neutralization schemes. The conductive coatings used in electron spectroscopy to analyze insulating surfaces would modify and fully obscure the chemistry of interest on a sample surface. Examples of thin surface enhancements on insulating samples will show that these can be immediately characterized as surface segregations, some of nearly monolayer thickness regions over large scales.

Multivariate methods are also finding foothold within the XPS community. As XPS is a spectroscopic technique at its heart, the high speed imaging afforded by modern systems allows stacks of data with both spatial and spectral information to be obtained. PCA processing of images will be seen to accentuates correlations while removing noise and cleaning up these images. The multivariate techniques can also be applied to the spectral components, fitting components to 64,000+ spectra generated at each pixel location from an image. These components are then used to generate a new image while removing noise, which is shown to improve the image quality.

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