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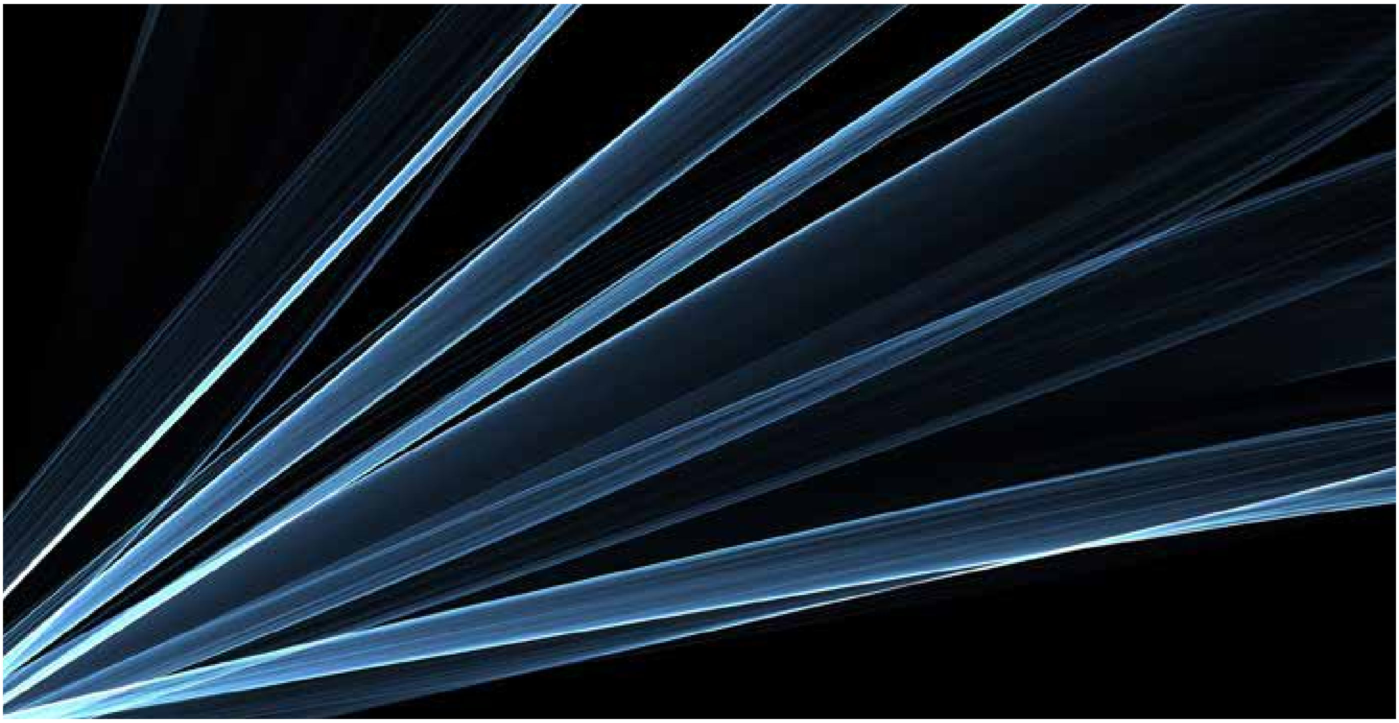
APPLICATION NOTE



X-ray Photoelectron Spectroscopy

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The chemistry and structure of a solid determine its inter-actions with other materials. In fields such as catalysis, adhesion, coatings and corrosion, the surface condition is critical, so advanced techniques for surface characterization are required. X-ray Photoelectron Spectroscopy (XPS) is a highly surface-sensitive, quantitative chemical analysis technique that can solve a wide range of problems. XPS is also known as Electron Spectroscopy for Chemical Analysis (ESCA).

Thermo Scientific XPS instruments can provide vital information on the surface of any solid or powder:

- Elemental Composition and Quantification
- Chemical State Identification
- Chemical State Quantification
- Lateral Distribution
- Depth Distribution

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Experimental

In an XPS experiment, the sample is illuminated with a beam of soft X-rays. These cause the ejection of photoelectrons from the atoms in the sample (Figure 1). A photoelectron spectrum is recorded by counting ejected electrons over a range of electron kinetic energies. Peaks appear in the spectrum from atoms emitting electrons of a particular, characteristic energy. The energies and intensities of the photoelectron peaks allow identification and quantification of all elements (except hydrogen) near the surface. Fine structure in the electron spectrum reveals variations in chemical bonding.

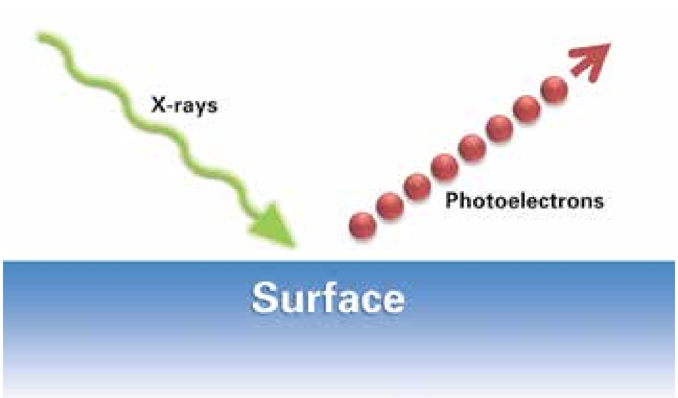

Figure 1: Ejection of photoelectrons

By varying the spatial resolution of the technique, it is possible to analyze small features, and to build up an image showing the distribution of elements and chemical states across the sample.

Inelastic scattering of photoelectrons occurs as they travel through the solid sample, so that they can lose their characteristic kinetic energy. This scattering effectively limits the depth through which photoelectrons can travel, and still be analyzed, to about 10 nm. This is where the surface specificity of XPS comes from. Fortunately, much of the important chemistry of a surface occurs within the top few nm, and XPS provides detailed information for that region.

Angle-resolved X-ray Photoelectron Spectroscopy (ARXPS) can be used to determine the thickness and composition of layers within this analysis depth without damaging the sample. For deeper analysis, XPS acquisition can be alternated with ion beam sputtering, to progressively remove material and build up a depth profile.

Both conductive and insulating materials can be studied with XPS. For insulating samples, charge neutralization systems allow high-resolution measurements to be made routinely.

Results

A patterned, fluoropolymer-coated sample was analyzed with XPS, to provide the following data examples.

Figure 2 shows a survey spectrum, scanned with high sensitivity across the entire electron energy range. The survey spectrum exhibits peaks from each element in the sample. The principal peaks are labelled, and peak areas are used to determine the quantities (in atomic %) of each element.

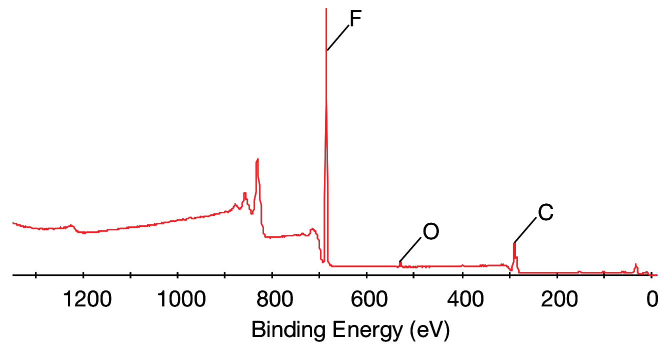

Figure 2: Survey photoelectron spectrum showing peaks from the elements at the sample surface, and quantification derived from peak areas

Figure 3 shows a high-resolution photoelectron spectrum, spanning just the main carbon peak. This spectrum was acquired from a fluoropolymer region of the sample. In this spectrum, there are multiple peaks due to a variety of bonds formed by the carbon atoms in the sample. Carbon atoms bonded to C, O and F will have different chemical shifts, so that their photoelectron peaks appear at slightly different energies. The different chemical states can be separated and quantified, providing important information about chemical bonding states at the sample surface.

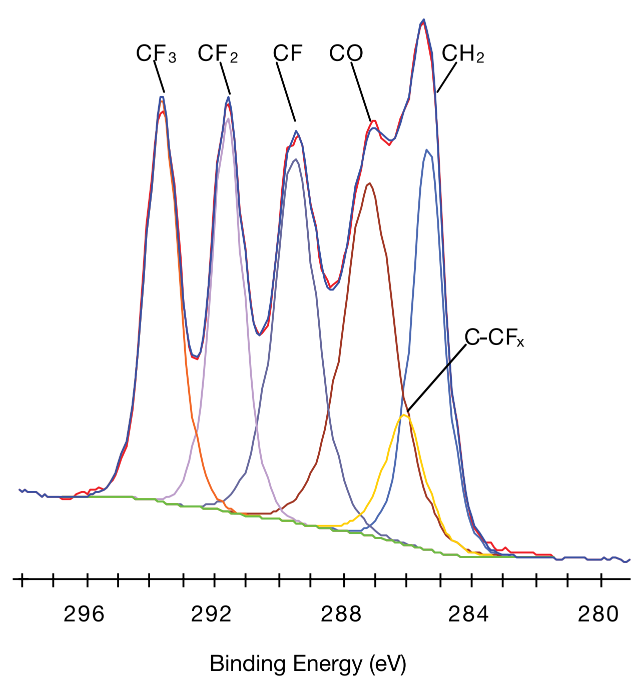

Figure 3: High resolution carbon photoelectron spectrum, showing six individual chemical bonding states of carbon

Figure 4 shows the results of an XPS imaging experiment, where the sample was moved in two axes while spectra were acquired using a highly-focused X-ray spot. By graphing the composition as a function of position we can easily construct a map of the sample, showing the pattern of chemical variations across the sample surface.

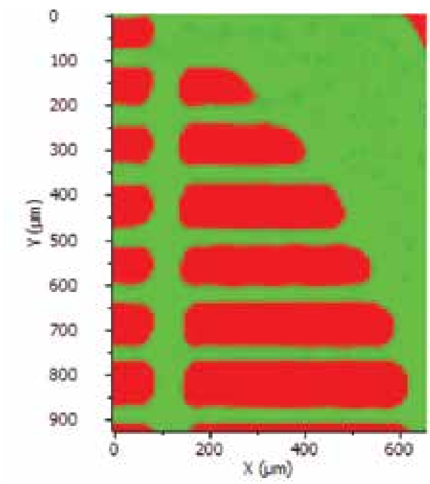

Figure 4: XPS map showing distribution of polymer substrate (green) and fluorinated coating (red)

Figure 5 shows a depth profile, obtained by acquiring XPS spectra in between low-energy argon ion beam sputtering phases. As thin layers of material are removed in each cycle, a compositional profile is constructed.

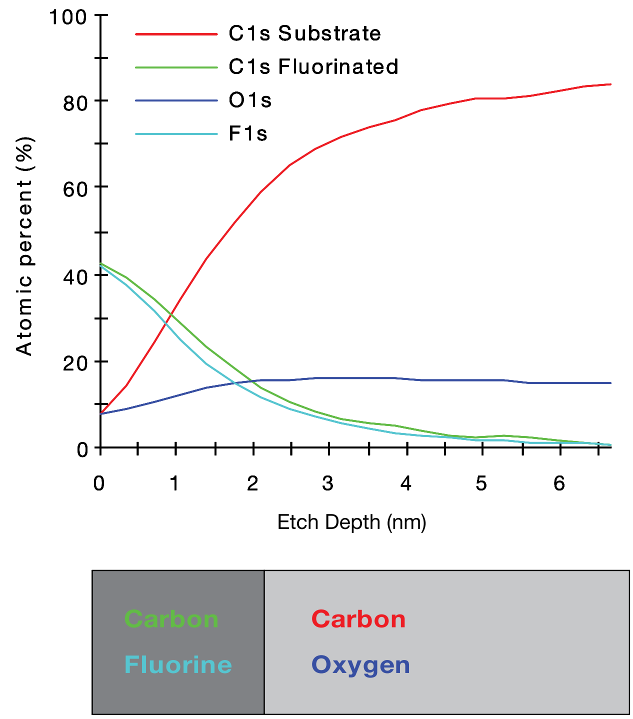

Figure 5: XPS depth profile showing the sample composition changing from a fluorinated surface layer to the unmodified polymer substrate. A schematic of the sample structure is shown below the profile.

Summary

X-ray Photoelectron Spectroscopy is a powerful probe of the surface chemistry of any material. XPS analyses are used to develop solutions in a wide range of applications, wherever surface conditions are important.

Thermo Fisher Scientific offers the most extensive portfolio of integrated XPS instruments, with unrivalled power, functionality and ease of use. The advanced Thermo Scientific Avantage data system enables comprehensive instrument control, acquisition, processing and reporting features, taking XPS from research to routine.

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Notes

Keywords
ESCA, Surface Analysis, XPS

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Find out more at **thermofisher.com/xps**

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