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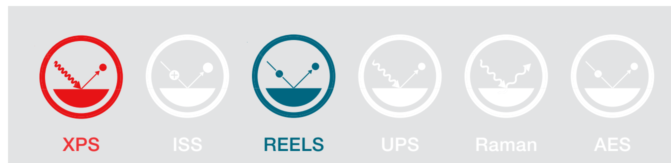
APPLICATION NOTE

Multi-technique analysis for surface hydrogen quantification

The Thermo Scientific™ ESCALAB™ Xi+ X-ray Photoelectron Spectrometer (XPS) Microprobe can be used to detect and quantify the presence of hydrogen in the near-surface region of a sample.



Introduction

A major strength of XPS as an analytical technique is its ability to provide quantitative information concerning the near-surface region of a solid sample. It is a limitation of the technique that the quantitative information excludes hydrogen.

This limitation means that up to two-thirds of the atoms in a polymer are ignored. In the case of an inorganic oxide surface, XPS alone might provide a misleading quantification if, for example, water or hydroxide is present at the surface. There are cases where knowledge of the hydrogen concentration is essential for development of the technology. An example of this is hydrogen storage materials for fuel cells.

This document will show that, by using the Thermo Scientific™ ESCALAB™ Xi+ XPS Microprobe, it is possible to measure the quantity of hydrogen within a few nanometers of the surface of a sample. Such measurements are available to all ESCALAB Xi+ XPS Microprobe users; there is no requirement to purchase additional equipment.

Experiment

The ESCALAB Xi+ XPS Microprobe has an electron source in its electrostatic lens column. It is necessary for it to be there so that XPS charge compensation can be accomplished when the magnetic immersion lens is being used. The position of this electron source is shown in Figure 1.

For charge compensation in XPS, this source is operated at a very low voltage (<1 eV). This source has an additional function in the ESCALAB Xi+ XPS Microprobe; it is used for physical, real-time, parallel imaging. For this reason, the source can be used to provide electrons whose energy is much higher (up to 1 keV). At the higher energies, this source can also be used for REELS (reflection electron energy loss spectroscopy).

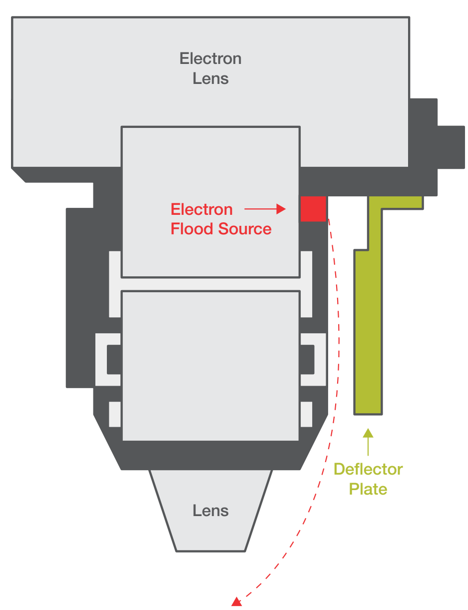


Figure 1: The position of the electron source used in these measurements.

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Figure 2 shows two REELS spectra, one from polystyrene and one from PFO (Poly (9, 9-dioctylfluorene)). Part of the large peak, due to the elastically scattered electrons, can be seen at close to 0 eV energy loss. The other peaks in the spectra are due to π–π* transitions within the aromatic molecules.

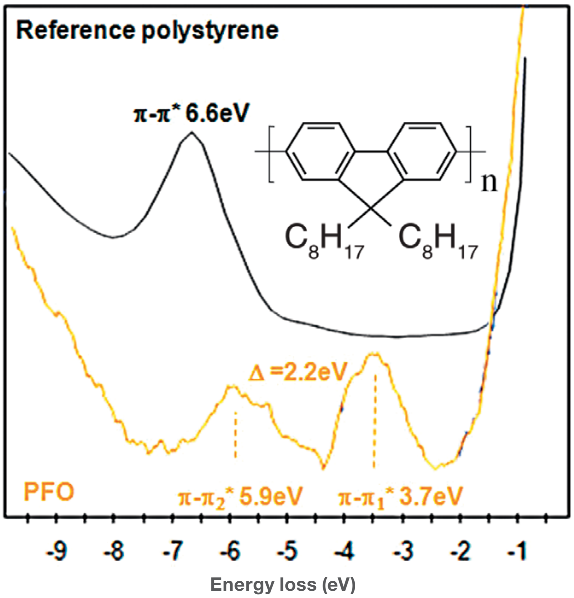

Figure 2: REELS spectra from two aromatic polymers showing energy loss features due to valence band transitions.

The inelastically scattered electrons can, therefore, provide useful information about the valence electrons in a sample.

Closer examination of the spectrum from the elastically scattered electrons shows that, in many cases, there is not just one peak but two. One peak is due to electrons elastically scattered from the hydrogen atoms in the sample, while the other is due to electrons scattered from all of the other atoms in the sample. This is illustrated in Figure 3, which shows REELS spectra from a series of polymers.

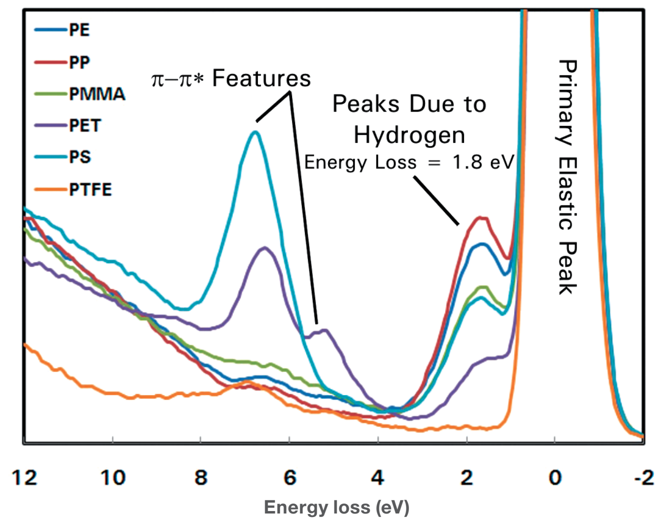

Figure 3: REELS spectra from a series of polymers.

High-density polyethylene (HDPE) shows the largest peak due to hydrogen, while other polymers show smaller peaks in line with the hydrogen content of each polymer.

Similar data can be obtained from inorganic materials, as can be seen in the REELS spectra from Al_2O_3, AlO(OH) and $Al(OH)_3$. See Figure 4.

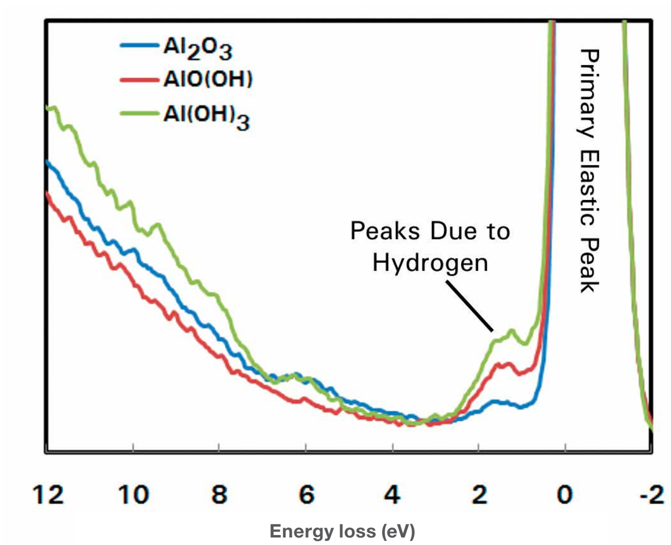

Figure 4: REELS spectra from aluminum compounds.

Using peak fitting techniques available in the Thermo Scientific Avantage Data System, the areas of the elastic peaks can be measured. Electron scattering cross sections are available (e.g. from NIST) and can be applied to the measured peak areas to arrive at a hydrogen concentration.

Results

This procedure was applied to the polymers whose spectra are shown in Figure 3 and others. The results are shown in Figure 5.

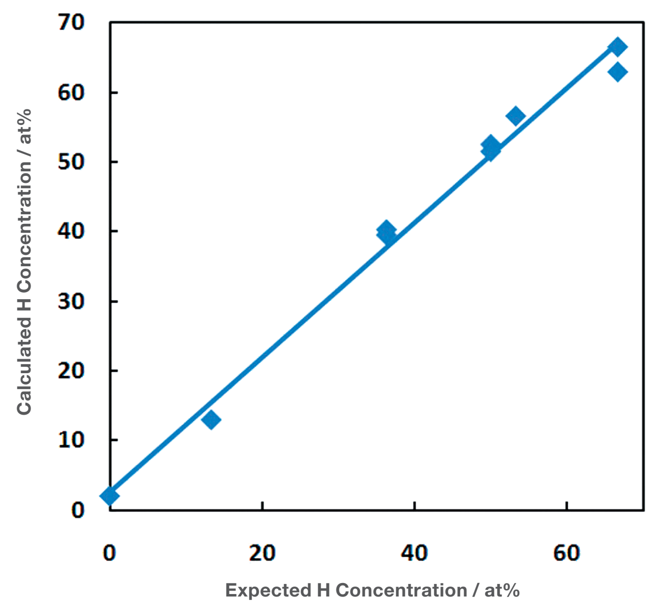

Figure 5: The measured hydrogen concentration plotted against the expected concentration.

As can be seen, there is good agreement between the hydrogen concentrations determined using REELS and the expected concentrations. For polytetrafluoroethylene (PTFE), there is a very small quantity of hydrogen present; this is likely due to contamination.

Similar calculations were applied to the aluminum compounds; the results are shown in Table 1. Again, there is good agreement between the measured and expected hydrogen concentrations. Absorbed water may account for the presence of hydrogen in the alumina sample.

Sample	Expected H Concentration at%	Expected H Concentration at%
Al_2O_3	0	9
AlO(OH)	25	29
$Al(OH)_3$	43	40

Table 1: Expected and measured hydrogen concentrations from three aluminum compounds.

Summary

The ESCALAB Xi+ XPS Microprobe has been successfully used to detect and quantify hydrogen on the surface of both organic and inorganic materials.

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Notes

Reference

http://www.nist.gov/srd/nist64.cfm

Keywords

ESCALAB XI+ XPS Microprobe, Hydrogen, Quantification, REELS, Surface Analysis, XPS

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