X-RAY PHOTOELECTRON SPECTROSCOPY

DESCRIPTION OF TECHNIQUE

X-Ray Photoelectron Spectroscopy (XPS), also known as Electron Spectroscopy for Chemical Analysis (ESCA), is an analysis technique used to obtain chemical information about the surfaces of solid materials. Both composition and the chemical state of surface constituents can be determined by XPS. Insulators and conductors can easily be analyzed in surface areas from a few microns to a few millimeters across.

The sample is placed in an ultrahigh vacuum environment and exposed to a low-energy, monochromatic x-ray source. The incident x-rays cause the ejection of core-level electrons from sample atoms. The energy of a photoemitted core electron is a function of its binding energy and is characteristic of the element from which it was emitted. Energy analysis of the emitted photoelectrons is the primary data used for XPS.

When the core electron is ejected by the incident x-ray, an outer electron fills the core hole. The energy of this transition is balanced by the emission of an Auger electron or a characteristic x-ray. Analysis of Auger electrons can be used in XPS, in addition to emitted photoelectrons.

The photoelectrons and Auger electrons emitted from the sample are detected by an electron energy analyzer, and their energy is determined as a function of their velocity entering the detector. By counting the number of photoelectrons and Auger electrons as a function of their energy, a spectrum representing the surface composition is obtained. The energy corresponding to each peak is characteristic of an element present in the sampled volume. The area under a peak in the spectrum is a measure of the relative amount of the element represented by that peak. The peak shape and precise position indicates the chemical state for the element.

XPS is a surface sensitive technique because only those electrons generated near the surface escape and are detected. The photoelectrons of interest have relatively low kinetic energy. Due to inelastic collisions within the sample's atomic structure, photoelectrons originating more than 20 to 50 Å below the surface cannot escape with sufficient energy to be detected.
ANALYTICAL INFORMATION

Survey Scan - Energy peaks in the survey scan identify the elemental composition of the uppermost 20 to 50 Å of the analyzed surface. All elements, except hydrogen and helium, are detected. Detection limits are approximately 0.1 atom percent for most elements.

High Resolution Multiplex Scan - This technique evaluates the chemical state(s) of each element through its core electron binding energies. Precise determination of binding energies are made through the use of curvefitting routines applied to the peaks in the multiplex scan. Shifts in the binding energy can result from the atom’s oxidation state, chemical bonds, or crystal structure. A NIST database is available to identify binding energies with specific compounds.

Quantitation - The concentrations of the elements identified in the survey scan are determined by integrating the area under a characteristic peak for each element. Sensitivity factors are applied to the peak area values to determine the elemental concentration.

Depth Profile - The elemental composition is measured as a function of depth into the sample by alternating AES analysis with ion sputtering to remove material from the sample surface. Depth resolution of <100 Å is possible.

Mapping - The relative concentration of one or more elements is determined as a function of lateral position on the sample surface. An image is obtained where brightness indicates the element concentration.

TYPICAL APPLICATIONS

- Analysis of thin film contamination
- Evaluation of adhesion failures
- Measurement of elemental composition of insulating materials (e.g., polymers, glasses)
- Identification of the chemical state of surface films (e.g., metal or oxide)
- Quantitative elemental depth profiling of insulators

SAMPLE REQUIREMENTS

Sample size cannot exceed 1 in. (25 mm) in any lateral direction. Height should not exceed \( \frac{1}{2} \) in. (12 mm). Sample must be compatible with an ultra-high vacuum environment (>10\(^{-9}\) Torr).