

The Information You Need...When You Need It.

## **Electron Spectroscopy for Chemical Analysis (ESCA)**

Electron Spectroscopy for Chemical Analysis, also known as X-ray Photoelectron Spectroscopy (XPS), provides information on the atomic composition and chemical bonding of a sample on the surface (top few nanometers). ESCA can detect all elements except for hydrogen and helium, and the detection limit is about 0.1 atomic percent.

We can use ESCA on both conductive and insulating samples, which mean this technique has a wide application for both organic and inorganic samples.

### **How it Works:**

In Electron Spectroscopy for Chemical Analysis (ESCA), we use an X-ray beam to bombard the surface of sample so we can measure the core electrons emitted. We have multiple types of excitation sources: a non-monochromatic Al or Mg x-ray and a monochromatic Al x-ray source. The choice of source is based on the sample under analysis and what information is needed about the sample.

The X-ray beam penetrates several microns into the sample. When this occurs, core electrons from the atoms of the samples are freed, but only the electrons from the near surface region (top few nanometers) can leave the surface. The instrument collects and measures the kinetic energy of these electrons from which we can calculate the binding energy, which originally held the electron to its source atom.

From the binding energy we can determine which element the electron came from and determine the oxidation state (chemical binding state) of that atom (for many elements).

An argon ion sputter gun can be used to remove the surface layers of the sample and monitor changes in elemental composition relative to depth. Since the escape depth for low-energy electrons is only 1-10 nm, ESCA is very surface-sensitive.

We also perform "small spot ESCA", which refers to the ability of the electron analyzer to aperture down to an area as small as 75 microns in diameter. When we combine this feature with a high-strength but small X-ray source, we are able to provide a chemical analysis of features smaller than 100 microns in width.

## **ESCA Applications Include:**

### *Materials Evaluation*

- Oxidation states
- Identification of polymeric coatings
- Surface compound identification
- Surface composition
- Plasma treatment
- Anti-reflection coatings

### *Failure Analysis*

- Corrosion product
- Material delamination
- Discoloration of epoxy
- Chemical degradation of surfaces
- Catalyst poisoning

### *Quality Control*

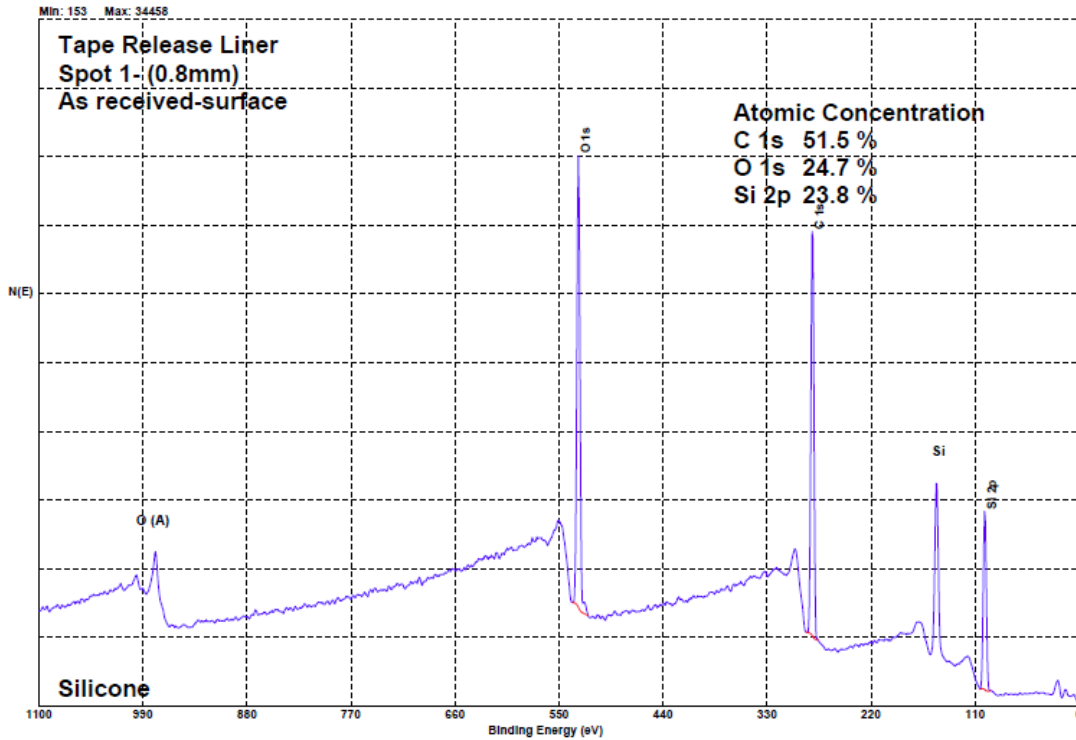
- Identification of organic contamination
- Breakdown of surface lubricants
- Chemical degradation of surfaces
- Adhesion failures

## **Data Presentation:**

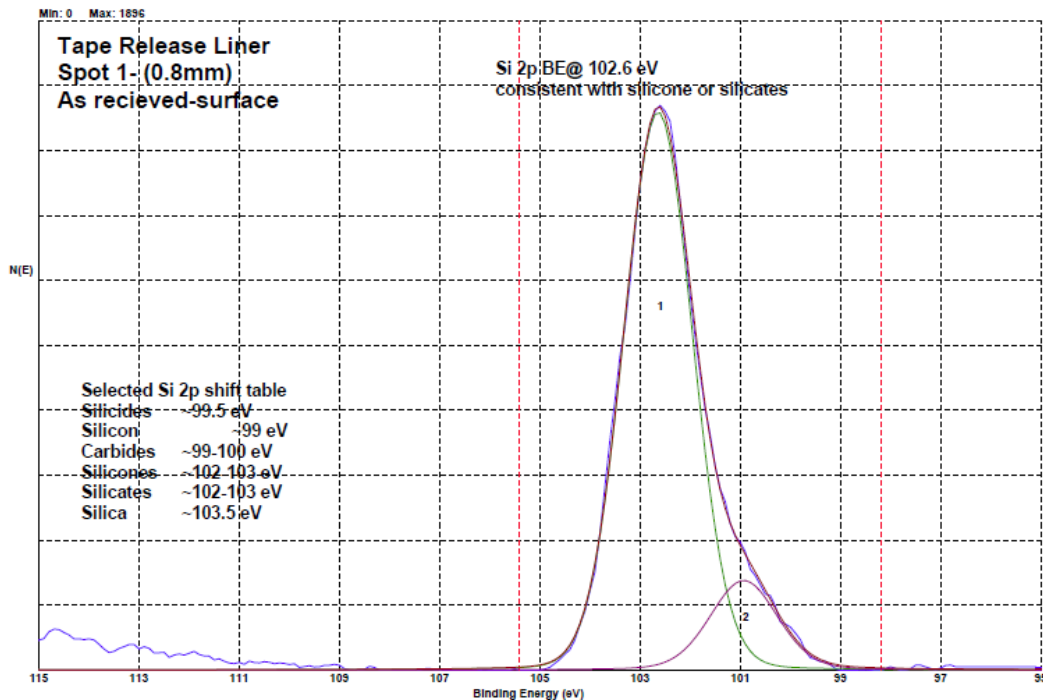
We usually plot ESCA spectra as the counts (number of electrons) versus electron binding energy. Using curve-fitting software, we can determine the oxidation state of the sample surface by interpreting binding energy shifts. The elemental composition of the sample surface can be determined by a "survey" spectrum, which usually includes a wide range scan from 0 eV to 1100 eV. Additionally, a "high resolution" spectrum can be used to discern subtle peak shape and peak energy changes. These high-resolution spectra usually cover a narrower energy range.

## **Sample Constraints:**

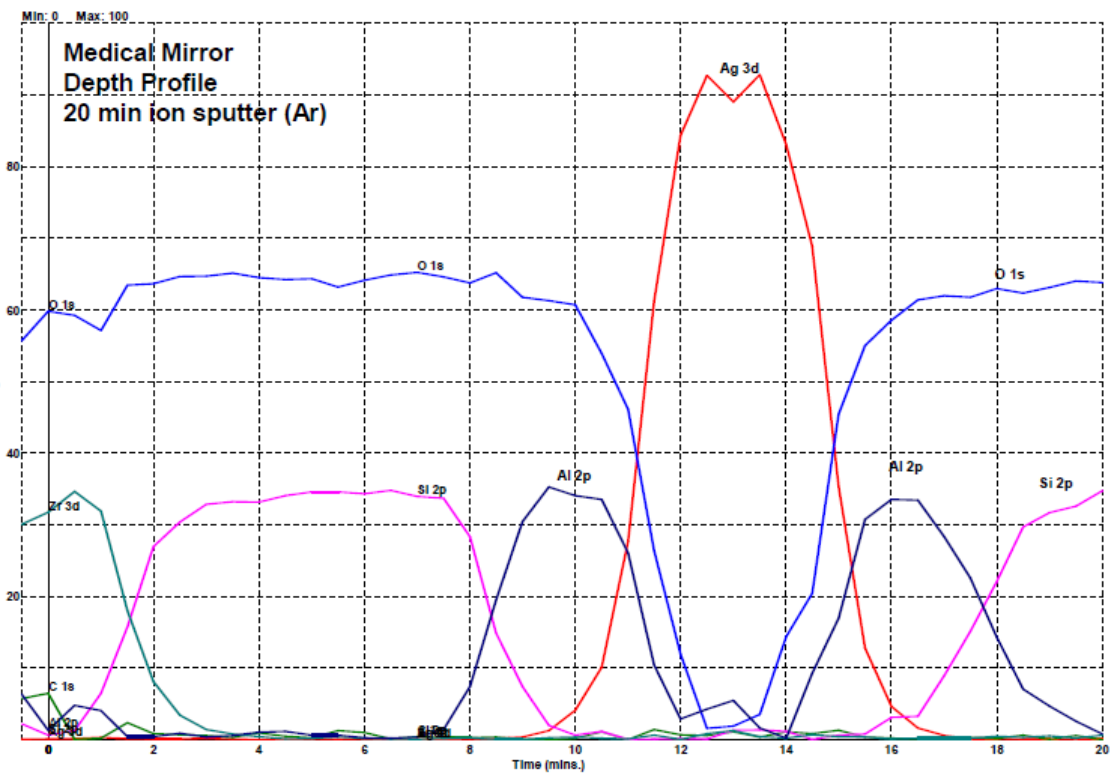
An ESCA sample can be up to 60mm in diameter x 13 mm high in size. We can analyze insulators, thin films, powders and organics. The sample must be non-volatile and be compatible with ultra high vacuum in the range of  $10^{-7}$  to  $10^{-10}$  Torr.



This is a typical "survey" or wide range scan. Scanning from 0-1100 eV it detects an element on the surface that is >0.1 At %. Ex: The surface of a Tape release liner has a silicone coating based on the 2:1:1 ratio of C:O:Si.



This is a high resolution scan which scans over a smaller range of binding energies and enables the detection of slight shifts in binding energies that normally represent different chemical states. Ex: A high-resolution scan of the silicon peak confirms silicones and a minor amount of another silicon state.



A depth profile can help one understand how the material changes as a function. Different materials have a different sputter rates so only approximate thickness can be assumed without standards. Ex: Above after a 20 minute profile the thin films of a medical mirror are zirconium oxide, silicon dioxide, aluminum oxide, silver, aluminum oxide and silicon dioxide.