TN 100

EAGLABS[SM] X-ray Photoelectron Spectroscopy/Electron Spectroscopy for Chemical Analysis (XPS/ESCA) Services

Technique Note

October 13, 2008 (Version 1.0)

XPS is a surface sensitive technique that can detect and quantify all elements except for H and He, and provide chemical state information, making it a powerful survey analysis technique

Introduction

X-ray Photoelectron Spectroscopy (XPS), also known as Electron Spectroscopy for Chemical Analysis (ESCA), is a surface analysis technique that is unique in providing chemical state bonding information. For example, it is widely used to determine localized bonding chemistry of carbon (i.e. C-C, C-O, C=O, O-C=O, etc.) and to differentiate oxidation states of inorganic compounds (i.e. sulfate vs. sulfide and metallic vs. oxidized states of metals). It is a quantitative technique and can detect all elements except for H and He with detection limits of approximately 0.1% atomic. XPS is an ultra-high vacuum surface analysis technique with a sampling volume that extends from the surface to a depth of 5-10nm. Its surface sensitivity makes XPS a technique of choice for analyzing thin contamination layers and characterizing outermost surface chemistries. XPS can also be utilized for sputter depth profiling to characterize thin films by quantifying matrix-level elements (>1% atomic) as a function of depth. The technique can accommodate insulating as well as conducting samples. The minimum area of analysis for XPS is approximately 30 µm.

Principles

The process works by irradiating a sample with monochromatic x-rays, resulting in the emission of photoelectrons whose energies are characteristic of the elements within the sampling volume. Furthermore, by detecting small shifts in the atomic binding energies of the photoelectrons, the chemical environments of the elements can be determined. Compositional depth profiling is performed with the aid of an ion beam that is used to sputter etch the sample between analysis cycles.

Common Applications

Its surface sensitivity, ability to analyze organic materials and other insulators, quantitative nature, and ability to provide chemical bonding information makes XPS an ideal technique for a wide variety of applications, including the following:

- Identifying stains and discolorations
- Characterizing cleaning processes
- Analyzing the composition of powders and debris
- Determining contaminant sources
- Examining polymer functionality before and after processing to identify and quantify surface changes
- Measuring lube thickness on hard disks
- Bonding and adhesion issues
- Obtaining depth profiles of thin film stacks (both conducting and non-conducting) for matrix level constituents
- Assessing the differences in oxide thickness between samples

Strengths

- Surface Sensitive; top 5-10nm
- Elemental identification of all elements except H and He
- Chemical state identification (oxidation state)
- Quantitative analysis
- Can analyze insulating as well as conducting samples
- Can analyze samples up to 8" in diameter and up to 1" thck
- Can analyze samples under cooled or heated conditions

Limitations

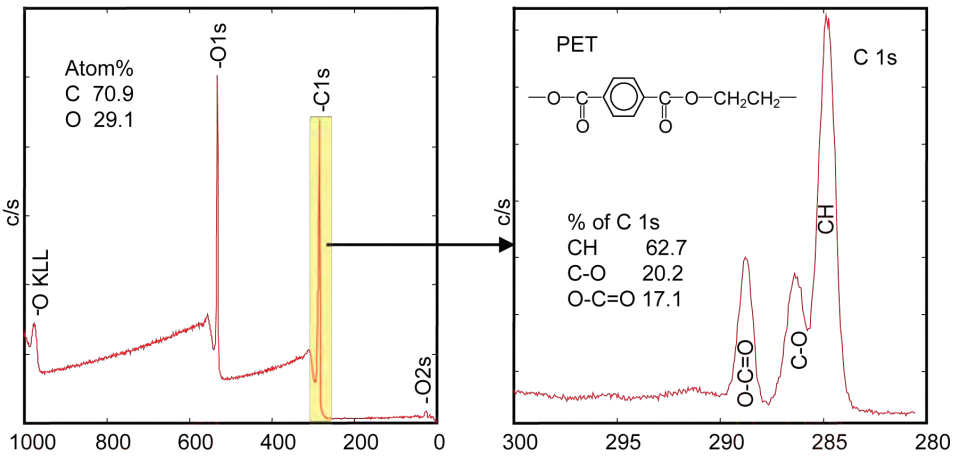
- Detection limits typically ~0.1% atomic
- Smallest analytical area ~30 µm diameter
- Limited organic information (short-range bonding only)
- Samples must be ultra-high vacuum compatible

Technique Comparisons

Other surface analysis tools with similar depths of analysis include Auger Electron Spectroscopy and Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS). XPS provides short-range chemical bonding information that is normally not obtained from Auger. TOF-SIMS provides complementary molecular bonding information. Insulating materials, including organic compounds, are routine for XPS and TOF-SIMS but are difficult with Auger. In terms of analytical beam sizes, Auger has the smallest spot size (~10nm), followed by TOF-SIMS (~0.3µm), and finally XPS (~10µm). XPS is considered a quantitative technique, Auger semi-quantitative, while TOF-SIMS results are very difficult to quantify other than for metal contamination on silicon.

Typical Data

Poly (ethylene terephthalate)



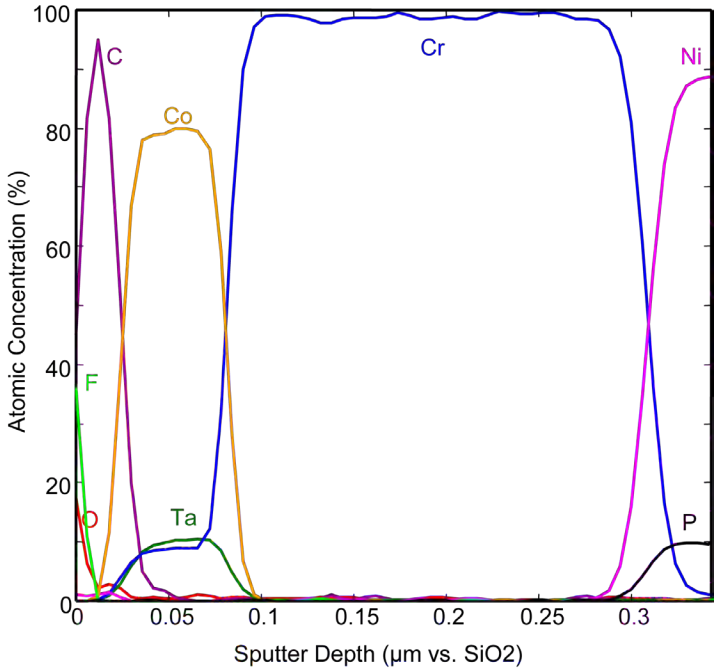
XPS survey spectra provide quantitative elemental information

XPS High resolution spectra provide quantitative chemical state information

XPS at Evans Analytical Group

EAG has 10 XPS instruments worldwide. Some of these instruments contain special capabilities such as an 8" sample stage for very large samples, a reaction chamber for custom-designed experiments, and a hot/cold stage for heating and cooling of samples in vacuum. Our XPS experience is unsurpassed with our XPS scientists having an average of over 14 years experience.

Magnetic hard disk



XPS sputter depth profiles provide composition with depth information