X-ray Photoelectron Spectroscopy

HOW DID WE GET HERE?

Discovery of X-ray

Wilhelm Röntgen, born in Germany in 1845, discovered X-ray in 1895 while performing his research on electrical rays at the University of Würzburg. This discovery earned him the Nobel Prize of Physics in 1901.

Analytical Equipment

Following almost 20 years of research at the University of Uppsala, Kai Siegbahn, born in Sweden in 1918, published a very detailed paper on XPS also known as "electron spectroscopy for chemical analysis" (ESCA). This work would be recognized by the Nobel Prize in 1981.

1887 — 1895 — 1905 — 1967 — 1969

Discovery of the Photoelectric Effect

In 1887, Henrich Hertz (Germany;1857), was studying electromagnetic waves propagation at the University of Karlsruhe. He observed for the first time the photoelectric effect when noticing the change of voltage required to create a spark between two metal electrodes after UV exposure.

Photoelectric Effect Theory

It is not until 1905 that the theory behind Hertz's observation would be formalized by Albert Einstein, (Germany;1879) while he was completing his PhD at the University of Zurich. He won the Nobel prize for that in 1921.

First Commercial Equipment

Following a collaboration with Siegbahn Hewlett-Packard introduced the first commercially available XPS in 1969.

HOW DOES IT WORK?

X-Ray Photoelectron Spectroscopy (XPS):

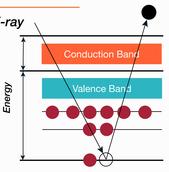
Why do engineers, medical doctors, and scientists need XPS?
For many industrial processes, surface contamination is the number one source of process variability. Cleanrooms exist to prevent dust and any other particles from coating semiconductor components. Surgeons scrub thoroughly to prevent germs and dust from being on their hands. Surface scientists sputter or plasma clean to improve their depositions onto their substrates. How clean a surface is, has a large impact on the quality of delivered products.

As new technologies such as 5G enter production, thin films and their interfaces are having more impact on the device properties than ever before. Measuring film composition, and chemical bonding states are pivotal to continue to drive innovation in the semiconductor industry.

XPS serves many industries in addition to semiconductors. It is widely used for both organic (bio and medical) and inorganic (semiconductor, photovoltaic, and LED) materials for new development, process control, and failure analysis. XPS is also a strong tool for the study of specialty materials like carbon nanotubes and graphene.

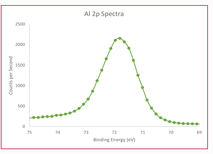
At the highest level, XPS serves as a chemical fingerprinting technique to identify surface atoms along with their chemical bonding states. When coupled with Argon (Ar) sputtering, XPS can destructively measure composition and chemical bonding as a function of depth. We will go through a few of the basic details followed by brief introductions to more advanced XPS modes.

Energy Level Diagram of XPS Process



The basic XPS setup is as follows: X-ray (high energy light source) is directed to the sample surface and cause photoelectrons to be emitted. Photoelectrons are energized electrons that are emitted from the surface when it is exposed to X-ray whose energy is above the threshold energy. Those photoelectrons are then captured, and their energies (the difference between the X-ray energy and the binding energy) are measured to determine which type of elements are present on the surface.

Every element on the periodic table has a different number of electrons and the easiest electron to remove is the outermost one. Energy level for each electron vary by electron orbital and the chemical bonding of the sample. These basic concepts support the fact that there is a semi-unique fingerprint for every element in the periodic table. Some peaks overlap in energy between elements but it is usually still possible to determine elemental composition.



Reading XPS Spectra

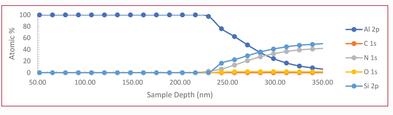
Typical XPS spectra have counts on the y-axis which represent the number of electrons impacting the detector. The relative height differences that trace out peaks is important, not the absolute counts. From a software side, the measured peak height and position are compared with a library of reference spectra to identify the material present. Most elements can be detected down to 0.1 atm % with a strong X-ray source or repeated sweeping counts.

APPLICATIONS

Chemical Bonding Information by Peak Deconvolution

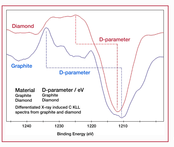
Sometimes XPS spectra contains overlapping signals, these are evident with asymmetry in peak shape, double peaks or long shelves. To extract the most meaningful data from these overlapped spectra a process known as deconvolution is used. Deconvolution is a mathematical approach that uses individual peaks of known shape to fit the larger composite peak. Deconvolution along with library of known XPS peaks for chemical bonding states, allows for the individual peaks that makes up the composite peak to be separated. In the above example, the measured carbon peak at 284.25 eV is refined into its component peaks at 284 eV, 285 eV and a small peak at 286.5 eV which represents different chemical bonding states. With a good library of peak positions and a high intensity X-ray source, XPS can give powerful insights into materials.

Depth Profiling by XPS



XPS is typically performed in a vacuum environment to minimize the chance of the photoelectrons colliding with anything other than the detector. Most systems are also outfitted with a sputtering beam, typically Ar, to clean the surface and perform XPS as a function of depth. This is useful if the layer of interest is not directly at the surface. X-rays penetrate deep into materials ~1 µm but only electrons emitted near the top 10 nm surface make it to the detector. By coupling XPS with the sputtering Ar ion beam, XPS can generates depth profiles of chemical composition and bonding states which is a very powerful information to understand thin film materials and their interfaces.

XPS for Graphene



The XPS spectrum contains Auger signals at high binding energy, which can be used for better understanding of the sample materials. In case of carbon material, XPS spectrum generate an Auger peak near 1225 eV. The D-parameter value (peak to valley distance of carbon's Auger peak) is well known for diamond (sp3) and graphite (sp2), the measured D-parameter of the sample material the ratio of sp3 and sp2 bonding states can be estimated.

CUSTOMER SERVICE WORKFLOW

STEP 1	STEP 2	STEP 3	STEP 4
Consult with Our Technologists	Provide Your Samples	Receive In-Depth Report	Debrief with Our Technologists

WHY CHOOSE OUTERMOST TECHNOLOGY FOR XPS?

1. State-of-the-art XPS tool for fast aquisition of high quality data
2. Elemental peak deconvolution for chemical bonding states
3. Integrated reports with analysis of measured data
4. Seamlessly combined with other metrology such as SIMS, Auger, RBS, nano-FTIR, and others)
5. Industry's most affordable pricing