Hard X-ray Photoelectron Spectroscopy for Multi-Layer Semiconductor Samples

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INTRODUCTION

Over the past decade, improved instrument reliability and automation has led to the broader use of X-ray Photoelectron Spectroscopy (XPS); changing from a specialist technique only performed by dedicated practitioners to a routine analysis tool that is widespread throughout academia and industry.

In typical operation, with the use of a monochromated aluminium anode, XPS has an inherent information depth of ~10nm. The most common methodology for determining the chemical composition further within the bulk is to destructively sputter away layers of material, exposing the underlying substrate, allowing a profile of the chemical composition with substrate depth to be generated through iterative cycles of sputtering and spectra reacquisition.

An alternative, non-destructive approach is to use incident radiation of a different energy, such as the higher energy, 'hard' X-rays, than that which is typically used in laboratory instruments to perform XPS (HAXPS). Whilst this approach has been used with great success at synchrotron facilities it has not yet found widespread use in laboratory XPS instruments.

A monochromated aluminium anode source is often the most commonly used X-ray source on systems, however other monochromated energies are available.

Non-monochromated sources have been available for many years, but often monochromated sources are more preferable due to the fact that non-monochromated sources give inherently wider peaks and satellite features due to other X-ray emission lines. Using software techniques to remove these satellite features make a non-monochromated source much more useful for everyday analysis.

With the wide range of options of anode materials, a large range of X-ray energies are available. Such materials include copper, titanium, magnesium and chromium. The variety of energies requires an analyser that can detect photoelectrons up to higher energies than the typical aluminium energy (1486.6 eV), and can often be done on the same system. This gives three major benefits: The ability to observe more core lines from elements, the ability to prevent any overlaps between X-ray induced peaks and Auger peaks in the acquired data from aluminium, and the ability to observe deeper than the 10 nm that is typically visible when using an aluminium source.

EXPERIMENTAL

The Thermo Scientific ESCALAB Xi⁺ was used for all data acquisition. The ESCALAB Xi⁺ was fitted with a dual anode monochromator with both an aluminium and silver coating. A non-monochromated twin anode source was also fitted to allow acquisition of data using magnesium and titanium X-rays.

These four anodes provide a wide range of energies from the principal core lines (Mg: 1253.6 eV. Al: 1486.6 eV. Ag: 2984.3 eV. Ti: 4510.8 eV), and hence a wide range of analysis depths.

Figure 1. Thermo Scientific ESCALAB Xi⁺

Thermo Scientific's Avantage software allows satellite subtraction from spectra acquired using non-monochromated sources. This was used when acquiring data using the Mg and Ti anodes.

The satellite subtraction works by inputting the range of energies for the emission lines and their respective intensities. When used, the software finds the peaks and subtracts the different emission lines from the acquired spectrum.

A sample of SiO_2 on a Si substrate was analysed to observe the effects of the different anodes.

RESULTS

Initially the software subtraction routine was tested using data acquired using the Ti anode.

An Si2p spectrum was acquired from the SiO_2 on Si sample, which would show a large elemental peak at 99.4 eV, with a smaller peak due to the SiO_2 at 103.5 eV. However, upon first look at the spectrum, shown by the red spectrum in Figure 2, it looks as though there is a large component due to the SiO_2 at an energy of 105 eV.

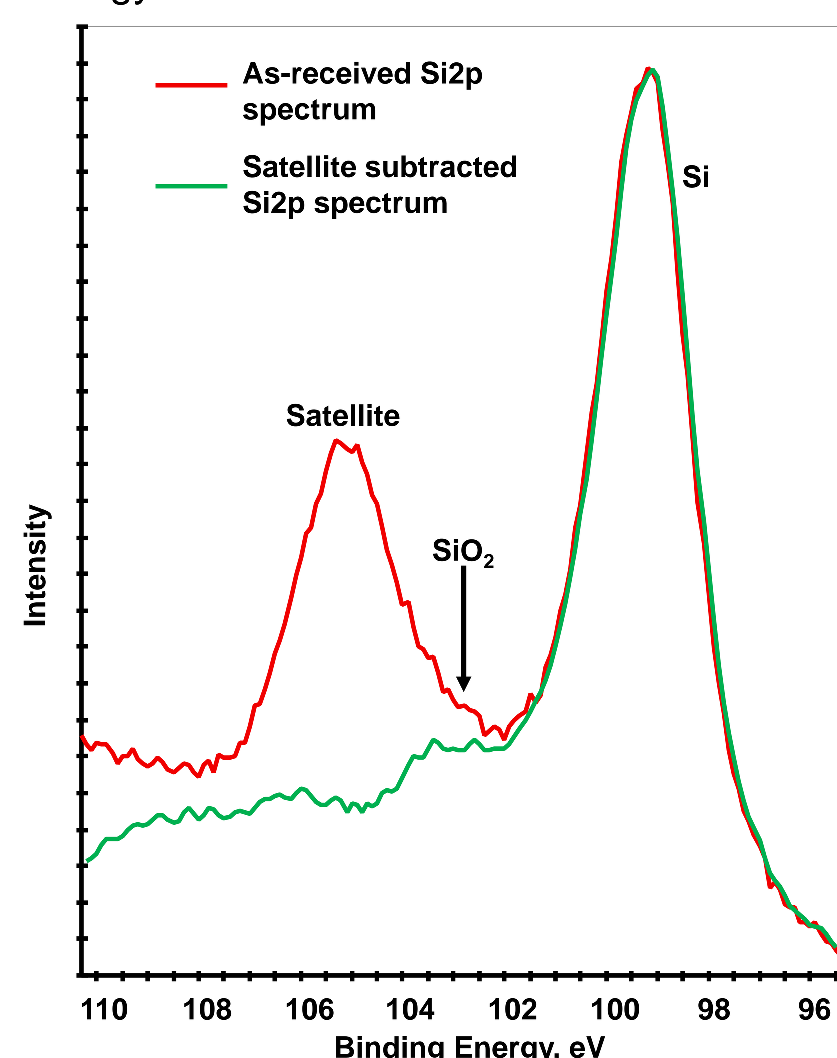

Figure 2. Comparison of the as-received and satellite subtracted Si2p spectra

However, this peak is due to a satellite from a secondary emission of Ti Kα_2, at an energy of 4,504.9 eV, or 5.9 eV lower than the main emission line with an energy of 4,510.8 eV.

Using the software, the satellite peak was subtracted. The spectra in Figure 2 show the pre-processed and post-processed Si2p spectra.

Data from the SiO_2 on Si sample was then acquired using the three other available anodes. The four as-received spectra can be seen in Figure 3. This includes the satellite subtraction on both the titanium and magnesium non-monochromated sources.

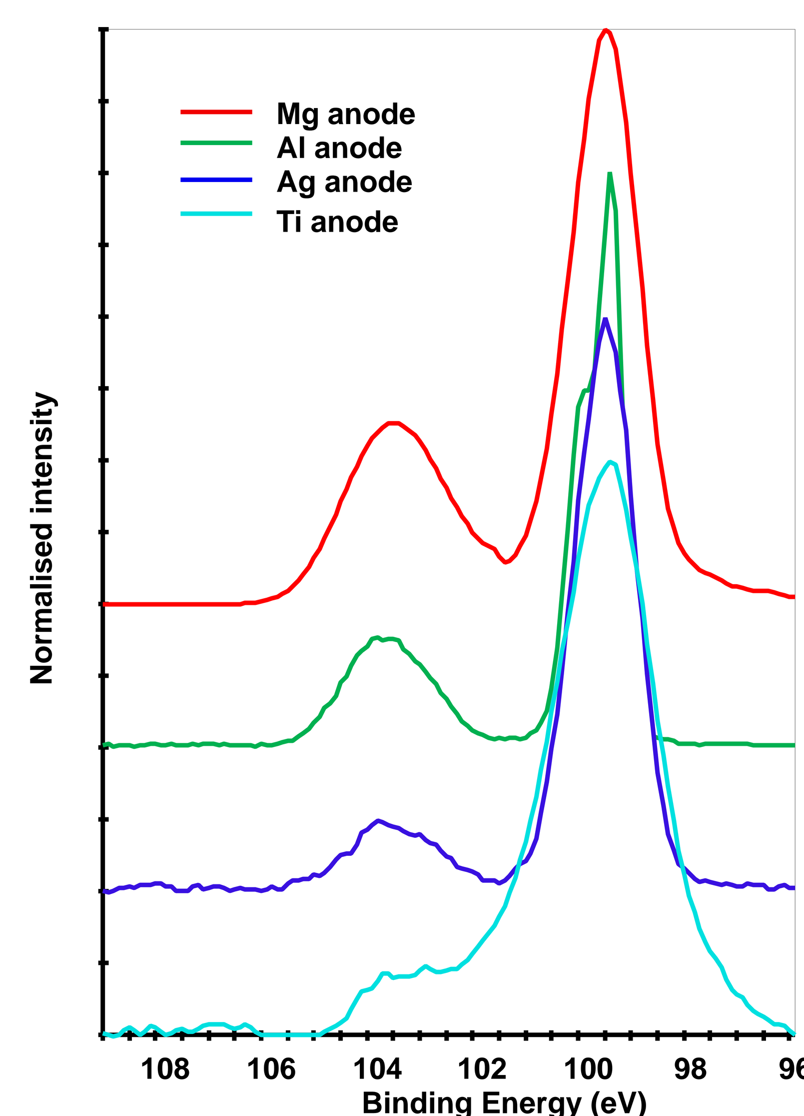

Figure 3. Comparison of Si2p spectra using different anodes

As expected, the higher the energy of X-ray, the lower the contribution of the SiO_2 peak compared to the elemental peak.

Table 1 shows the effect of the atomic percent calculation by using these different X-ray energies. Using the lowest energy, magnesium (1256.6 eV), gives 32.2% of SiO_2. Using titanium gives a value of only 4.4%.

Anode energy (Type), eV	Atomic percent	
	SiO_2	Si
1253.6 (Mg)	32.2	67.8
1486.6 (Al)	29.8	70.2
2984.3 (Ag)	14.7	85.3
4510.8 (Ti)	4.4	95.6

Table 1. Anode energy against atomic percentage of SiO_2 and Si substrate

Having four anodes, with the ability to use the software to remove any satellite peaks allows analysis of the sample into a variety of depths, without the need to profile the sample, and remove layers using an ion source.

Using the Single Overlayer Calculator within the Avantage software we can determine the attenuation length of the different anodes using the equation shown in Equation 1, which is derived from the Beer-Lambert law.

$$\frac{1}{\cos\theta} = \frac{t}{\lambda \ln[1 + K.R]}$$

Equation 1. "Single Overlayer Calculator" algorithm used to determine the attenuation length (λ)

Where t is the thickness of overlayer, θ is the emission angle, λ is the attenuation length, K is the ratio of atomic densities (element / overlayer) and R is the measured intensity ratio (overlayer / element).

Once calculated for each X-ray energy we can plot this on a graph to see how the attenuation length (and hence the analysis depth) will change with energy, as shown in Figure 4.

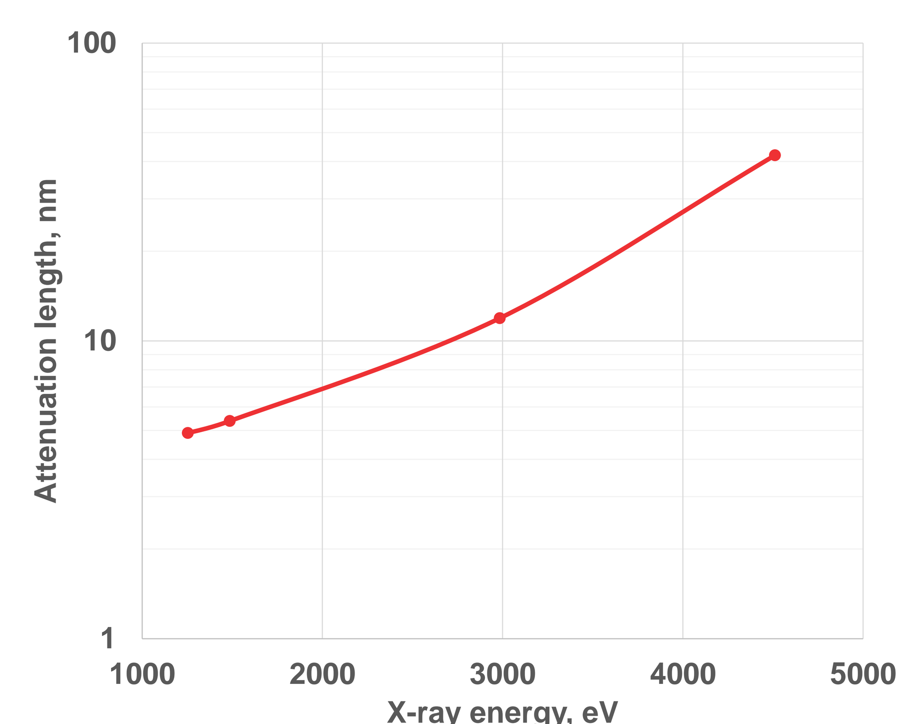

Figure 4. X-ray energy against attenuation length for silicon sample

This shows that a variety of anodes gives a wide range of analysis depths which have wide uses in multilayer samples.

CONCLUSIONS

By using several anodes which provide differing X-ray energies, it is possible to probe into different thicknesses of a sample, without the need to destructively etch it and remove material from the surface layers.

Using non-monochromated sources introduces secondary satellite peaks from different energies, but by using post-processing software satellite-removal techniques then using a non-monochromated anode makes analysing the data acquired as easy to analyse as data from a monochromated one.

REFERENCES

Determination of the inelastic mean free path (IMFP) of electrons in germanium and silicon by elastic peak electron spectroscopy (EPES) using an analyser of high resolution - Gergely, Konkol, Menyhard, Lesiak, Jablonski, Varga, and Toth