Quantitative Lateral Resolution of the Kratos AXIS Ultra and AXIS Nova XPS Instruments

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Small area X-ray Photoelectron Spectroscopy in the 10's of micron range has become an important tool for the surface analyst. Two approaches to producing small area XPS information are currently used in commercial XPS instrumentation. One approach employs a micro-focused X-ray probe to limit the sample area illuminated by X-rays during measurement. The second approach, utilised in the Axis range of spectrometers, is to use a system of electrostatic lenses and apertures to limit the acceptance area of the analyser. In both cases the analysed area on the sample surface is determined by measuring the distance required to receive a 20 to 80% signal increase when scanning the spot across a sharp edge. This is standard practice as described in the literature.[1]

Recent work by U. Scheithauer[2] has highlighted the fact that signal can be obtained over an area many times greater than the quoted spot size when recording XPS data using X-ray microprobe technology. This investigation utilises an approach similar to the aforementioned work to determine quantitatively the total area measured during small area XPS measurements using the virtual probe approach.

2 methods of generating small spot spectroscopic data:

Figure 1

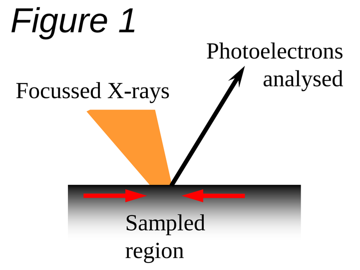


Figure 1: Focused X-ray beam with a small diameter to excite only a small, well defined area.

Figure 2

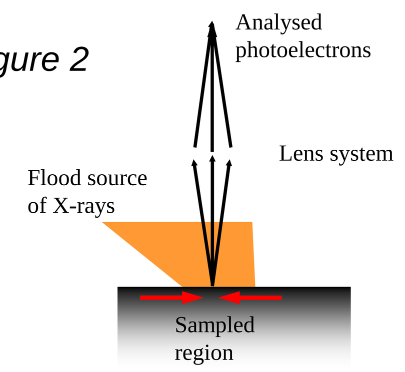


Figure 2: Broad 'flood' source of X-rays used to excite a large area, lens system limits the collection area.

Small Spot Spectroscopy on Axis Instruments

The Axis instruments use a combination of aperture, iris, magnet lens and electrostatic lens systems (Figure 3) to limit the analysed area on the sample surface.

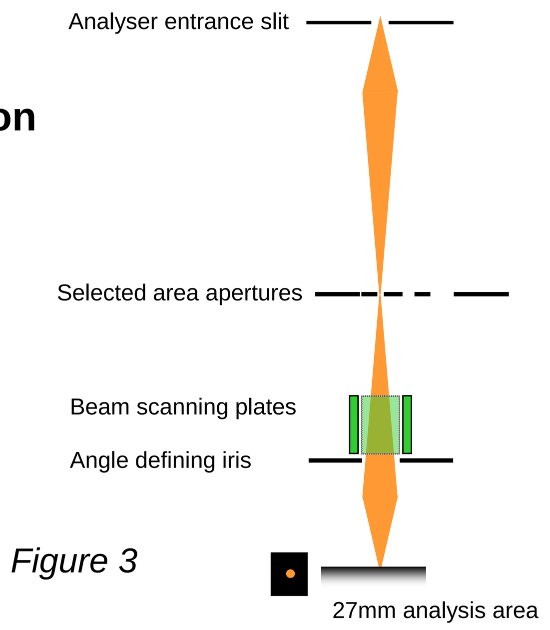


Figure 3

Definition of Spot Size

The international standard for spot size in XPS measurements can be considered to be the distance it takes for the signal intensity to change from 80% of maximum to 20% of maximum when the spot (focuses X-ray or lens defined area) is moved across a sharp edge, Figures 4 and 5 below.

Figure 4

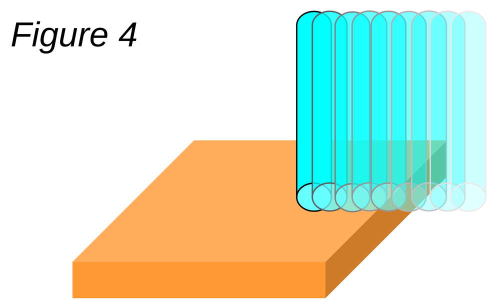


Figure 5

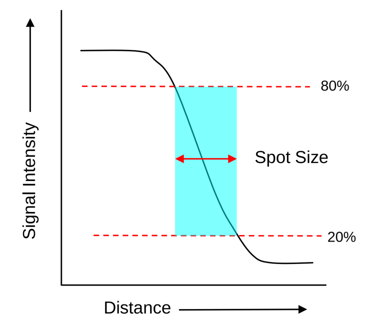
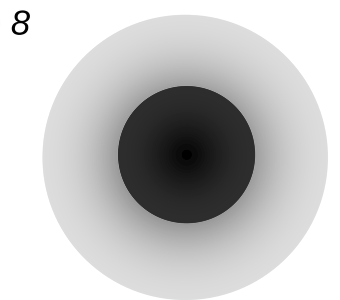
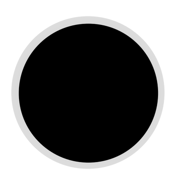


Figure 8



Wide iris makes aperture edges fuzzy

Figure 9



Closing iris sharpens up aperture edges

Effect of the Aperture & Iris

In the lens column of the Axis instruments there is a spot size aperture and an iris. The virtual photoelectron image of the sample surface is brought into focus at the aperture plane, therefore, by placing an aperture at this position the area analysed is controlled.

The iris controls the angular acceptance of the lens.

For every aperture size the iris setting is optimised to produce the desired analysis size on the sample. The effect of reducing the iris diameter on the 80/20 measured size of the 27 micron aperture is displayed below (Figure 6). The standard iris setting (red line, Figure 6) is 'sharpened' up by closing the iris (blue line, Figure 6). Figures 8 and 9 demonstrate the effect pictorially. However, the measured spot size is not greatly effected (Figure 7). The iris 'fine tunes' the size but the value reaches a minimum where it is un-affected by decreasing the iris more.

Figure 6

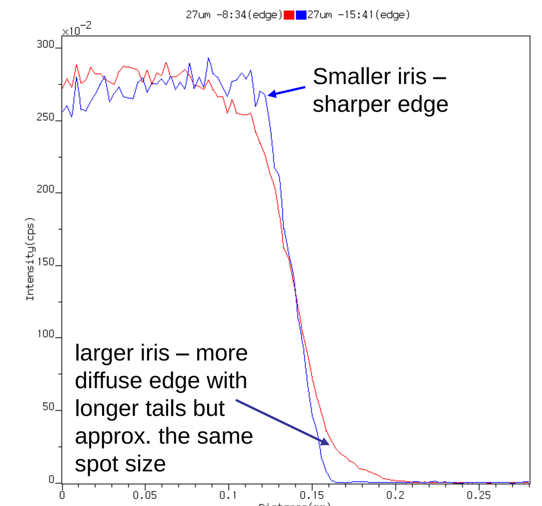
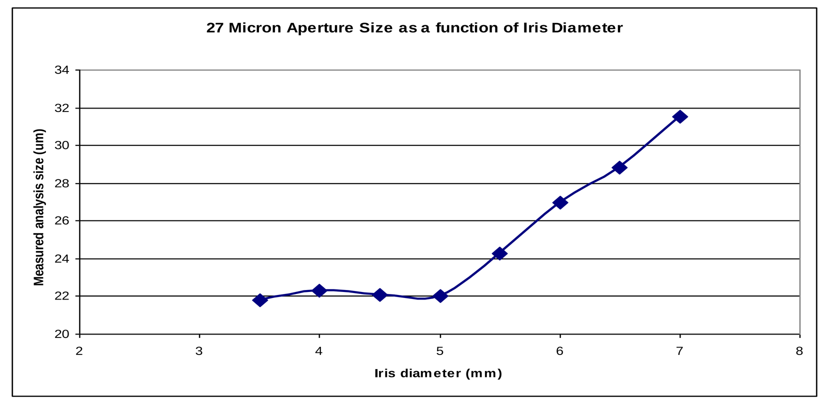


Figure 7



Total Analysed Area

In many applications it is important to know the maximum area that data is recorded from. An example could be looking for contaminants on bond pads. Do trace elements detected arise from surface bond pad contamination or are they from the substrate? To answer this type of question we need to consider the maximum likely analysed area at the spot size chosen. The 80/20 rule means that approximately 90% the signal will arise from within the area chosen but there is a significant tailing of the spot size which means that signal can be collected from well outside this defined area.[3]

To investigate quantitatively the amount of signal received from outside the specified analysis spot size a series of different sized 'holes' were analysed. These holes were purchased from National Aperture Inc. and consisted of holes of defined sizes in gold coated copper discs. The holes were mounted over screw holes in the sample holder so that no signal could arise from within the hole. Therefore, any signal detected when the small spot was positioned at the centre of the hole must have come from the surrounding surface. Hence, a simple ratio of the signal detected in the centre of the aperture and the signal detected when the small spot was placed on the Au surface allowed us to calculate the percentage of signal detected from an area outside the hole diameter, i.e. the percentage of signal that arose from an area with a diameter greater than the hole size.

An optical image of the 100 micron hole is displayed below (Figure 10) and a Au 4f parallel photoelectron image of the hole labelled with the analysed positions inside and outside the hole (Figure 11).

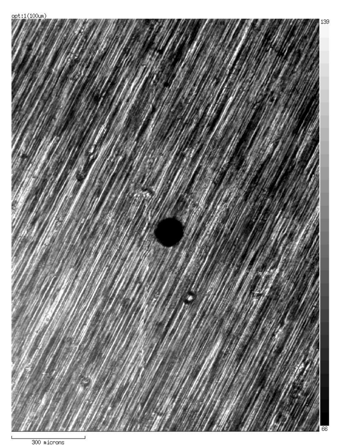


Figure 10: Optical image of the 100 micron hole

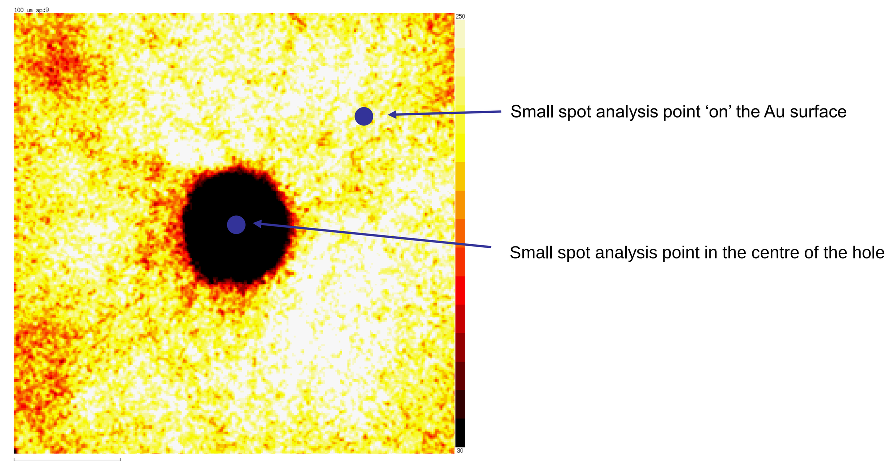
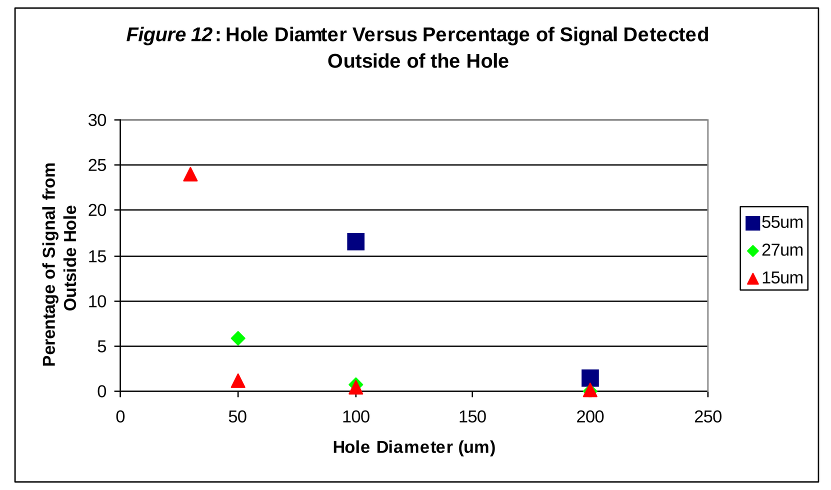
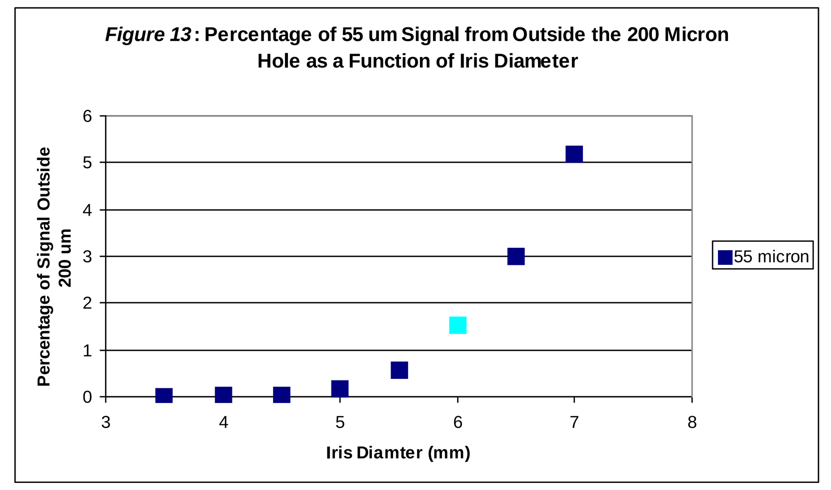
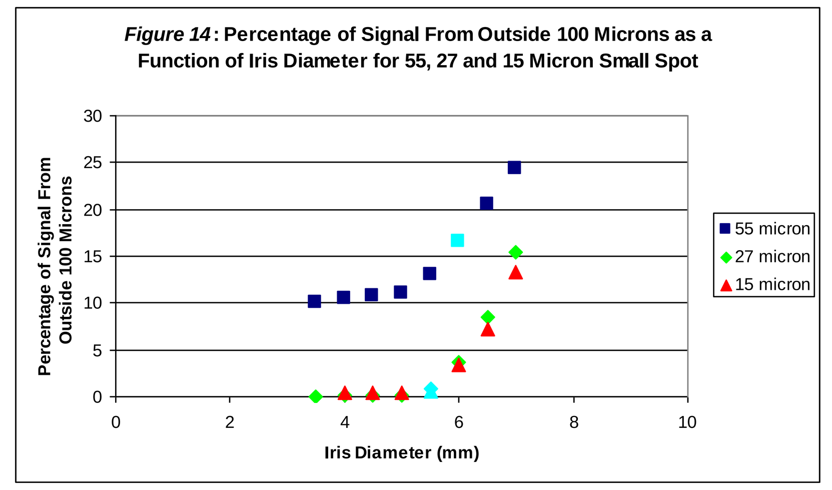


Figure 11: Small spot spectra were recorded from the centre of the hole and from the plain Au region.





Standard Iris Setting determined using the 80/20 specification



Standard 55, 27 and 15 micron Iris Setting determined using the 80/20 specification

The signal recorded (as a ratio to the signal from the substrate) from 55, 27 and 15 micron spot sizes positioned at the centre of several differently sized holes was measured. In each case the standard iris setting used to give the 80/20 spot size measurement was used. The percentage of the signal recorded outside the hole was plotted against different hole sizes for 55, 27 and 15 micron small spots (Figure 12). Surprisingly only spot sizes 27 and 15 microns showed negligible signal from greater than 100 microns. 16% of the 55 micron small spot signal arose from outside 100 microns and 1.5% of the 55 micron signal from outside 200 microns.

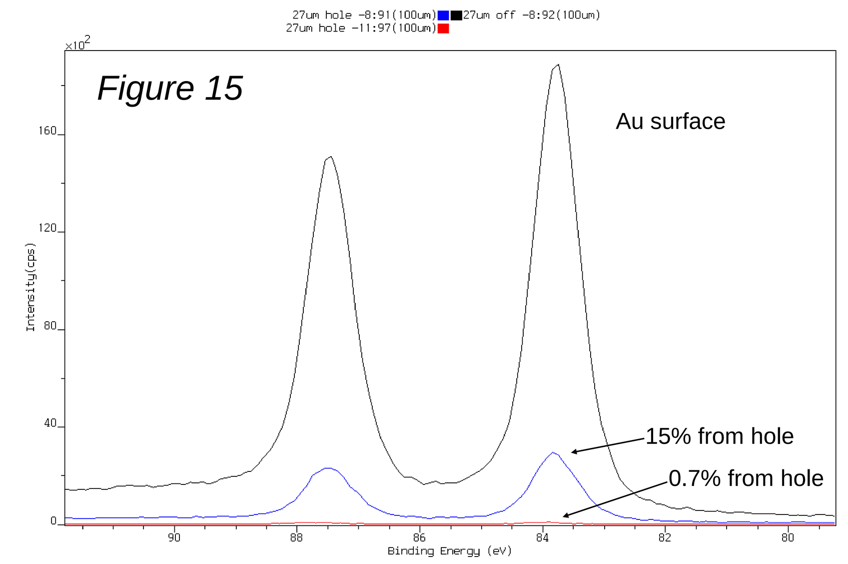
The effect of the iris diameter on the percentage of signal detected from outside the 200 micron hole was investigated for a spot size of 55 microns (Figure 13). By closing down the iris it was possible to reduce the signal to negligible values (less than 0.01%).

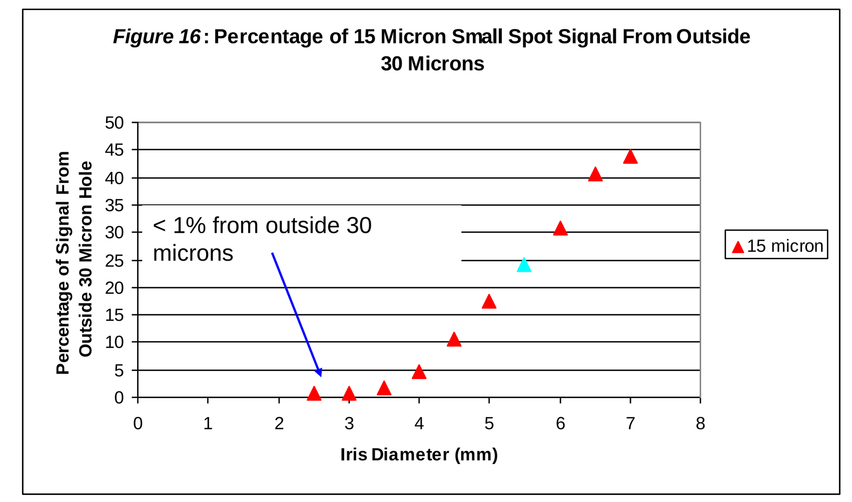
The 100 micron hole was then placed at the analyses position. The effect of iris diameter on the percentage of signal which arose from outside the 100 micron hole was then investigated for different spot sizes. The results are plotted in Figure 14 which graphs the iris diameter versus percentage of signal from outside the 100 micron hole when 55, 27 and 15 micron small spots were placed in the centre of the hole.

Figure 15 compares the Au 4f spectrum obtained from the sample surface (black) and from the centre of the 100 micron hole with different iris diameters (blue = 7 mm, red = 5.5 mm). By reducing the iris diameter the 27 micron spectrum from the hole is reduced to negligible size.

Reducing the hole size to 30 microns allowed the investigation of the 15 micron spot size. The 15 micron small spot was placed at the centre of the 30 micron hole and the percentage of signal detected from outside the 30 microns plotted against the iris diameter (Figure 16). Results indicate that a significant amount of the signal is detected from an area outside 30 microns when the standard iris setting is used (24%) but this can be reduced to less than 1% by reducing the iris diameter.

Figure 15





Standard Iris Setting determined using the 80/20 specification

The effect of the iris on the total analysed area was demonstrated by recording small spot spectra from a bond pad (spectra displayed below). In this example a 27 micron small spot was recorded from a 90 micron Pt bond pad on a Si substrate (Figures 19 and 20). Si was detected on the Pt pad but was this from the substrate or a surface contaminant of the pad? Without the control of the total analysed area provided by the iris system this question would be very difficult to answer. However, by closing down the iris we eliminated signal from the surrounding substrate and enabled unambiguous analysis of the pad (Figure 20).

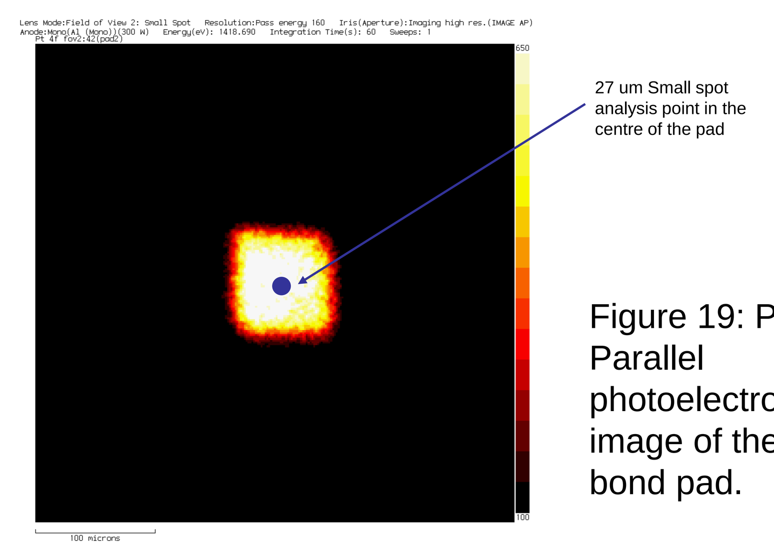


Figure 19: Pt 4f Parallel photoelectron image of the 90 um bond pad.

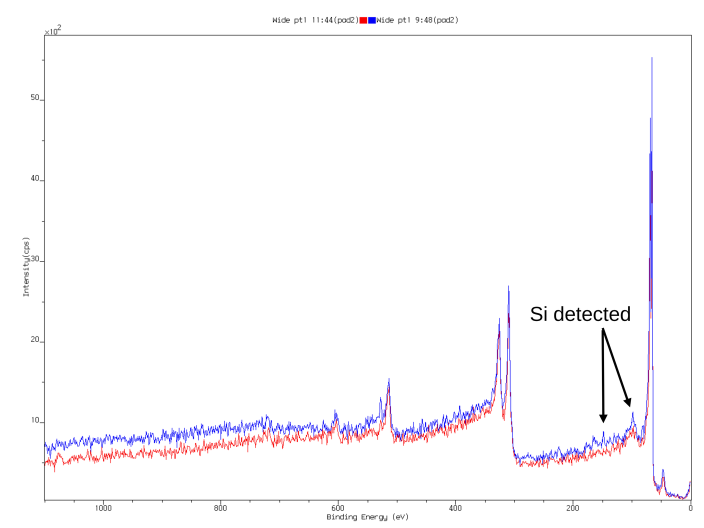


Figure 20: Overlay of 27 micron spectra from the centre of the bond pad with the iris open (6.5 mm - blue) and closed (5.5 mm - red).

Atomic concentration	Si	Pt
Iris Open	29.2	70.8
Iris Closed	0	100

Quantification results show that a significant amount of Si was detected with the iris open.

Conclusion

A surprisingly large percentage of the signal recorded during small spot spectroscopy originates from well outside the specified spot size when the spot size has been determined using the 80/20 rule. Signal can be detected from more than 3 times the quoted spot size. However, the total analysed area can be effectively controlled by closing down the iris on Axis spectrometers. It would appear that the use of an angle defining iris in the lens column is most beneficial for selected area spectroscopy.

[1] ISO 18115:2001.
[2] U. Scheithauer, Surf. Int. Anal., **40**, 706-709 (2008).
[3] D.R.Baer, M.H. Engelhard, Surf. Int. Anal., **29**, 766-772 (2000).