Application Note: 31054

Alpha110: Hemispherical Electron Energy Analyzer

Key Words

- Surface Analysis
- AES
- ARXPS
- ISS
- SAXPS
- UPS
- XPS

Introduction

The Thermo Scientific Alpha110, Figure 1, is a compact, high-performance analyzer for surface analysis applications, featuring true 180° geometry.



Figure 1: The Alpha110 analyzer

Features

- Energy resolution < 5 meV
- High sensitivity for XPS, UPS, AES and ISS
- Small area XPS capability
- Angle resolved XPS capability
- Compact design
- Avantage Data System

Description

Key parts of the analyzer include:

- 110 mm mean radius hemispherical analyzer
- Full 180° deflection
- Alpha plates at the analyzer entrance to optimize energy resolution
- Four pairs of slits to control energy resolution
- Multi-element input lens
- Flange-mounted 7-channeltron detector
- Mu-metal casing provides good performance at low kinetic energy, eliminating the need for internal shielding
- Full computer control

The Transfer Lens

The large acceptance angle of the lens ensures high sensitivity, while the slim profile ensures that there is the minimum risk of mechanical clashes with other components present in the same vacuum system.

The optimum working distance of the lens is 23.5 mm but it can be refocused to allow for a wide range of working distances.

The lens contains a twin-aperture system, used to define the analysis area and the angular acceptance of the lens. It is operated with a single rotary control and the principle of its operation is shown in Figure 2.

There are 6 aperture combinations on the mechanism; large area, 600 μ m, 350 μ m, 200 μ m, 100 μ m and the angle resolved setting. On the angle resolved setting, the angular acceptance is ±1°.

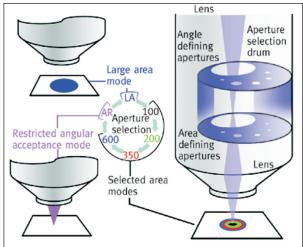


Figure 2: The operation of the twin aperture system on Alpha110



The Analyzer

Alpha110 is a true 180° hemispherical analyzer having a wide gap between the two hemispheres to accommodate an array of seven channeltron detectors. The multi-channel detector system provides high sensitivity coupled with a large dynamic range.

The analyzer is fitted with a set of four slits, controlled by a single rotary drive, allowing selection of the energy resolution. The ultimate energy resolution of the Alpha110 is < 5 meV.

The formula below shows that the energy resolution of the analyzer, ΔE , is not just dependent on the slit width, W, mean radius, R, and the pass energy, Ep, but also on the acceptance angle, α .

$$\Delta E = E_p \left(\frac{W}{2R} + \frac{\alpha^2}{2} \right)$$

Alpha-plates are fitted at the entrance to the analyzer to limit the acceptance angle for optimum energy resolution.

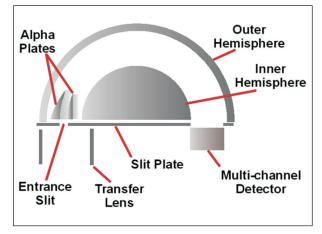


Figure 3: Analyzer cross section showing the position of the alpha-plates

The use of alpha-plates minimizes peak tailing, ensuring more accurate peak fitting and quantification of multi-component peaks. A further benefit of alpha-plates is improved field termination at the entrance plane. Poor field termination will adversely affect signal intensity.

The vacuum housing is manufactured from mu-metal, ensuring excellent magnetic shielding which is essential for high sensitivity to electrons of low kinetic energy. There is no need for additional internal or external shielding.

Electronics

The digital electronics package is fully integrated with the *Avantage* data system. It provides all lens, analyzer and detector voltages under data system control. In addition, the electronics are bi-polar to allow ISS measurements to be made.

X-ray Photoelectron Spectroscopy

Intensity Measurements

When reporting specifications for Alpha110, intensity measurements are dependent on source, sample and input lens geometry. We use a source to input lens angle of 55°, (the 'Magic Angle'), which is optimum for consistent quantitative analysis as asymmetry effects in this orientation are minimized. The sample is positioned so that the surface normal is aligned with the lens axis.

For selected area analysis, the sample is placed normal to the input lens to ensure a uniform, symmetrically shaped projection into the analyzer. Again, the source to input angle is 55° to minimize quantitative asymmetry factors.

Energy Resolution

Figure 4 shows the normalized spectra collected using Mg K α from the XR3 twin-anode X-ray source, operated at 300 W. The angle between the analyzer lens and the X-ray source was 55° when these data were collected.

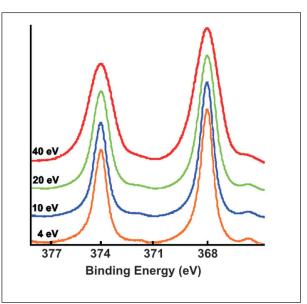


Figure 4: Normalized Ag 3d spectra from the Alpha110 analyzer at a series pass energies. Data acquired using non-monochromated Mg K α radiation.

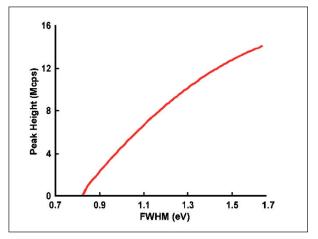


Figure 5: Ag $3d_{5/2}$ peak intensity as a function of resolution for the spectra shown in Figure 4

Figure 5 shows the relationship between the peak height and the full width at half maximum (FWHM) for the spectra shown in Figure 4. In these spectra, the ultimate energy resolution using the non-monochromated X-ray source is < 0.82 eV.

Improved energy resolution is obtained using the XR5 X-ray monochromator, as can be seen in the Ag 3d spectra in Figure 6.

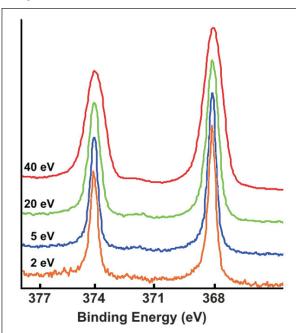


Figure 6: Ag 3d spectra acquired using the XR5 X-ray monochromator and the Alpha110 analyzer

Peak intensity as a function of peak width is shown in Figure 7 where the ultimate energy resolution is < 0.46 eV.

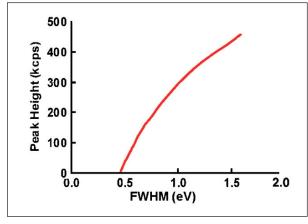


Figure 7: Ag $3d_{5/2}$ peak intensity as a function of resolution for the spectra shown in Figure 6

The natural line width of the Ag 3d peaks determines the minimum peak width in these spectra. Figure 8 and Figure 9 show data achieved from a cleaved sample of MoS₂. In this material, the natural line width is smaller, allowing Alpha110 to demonstrate a FWHM for the Mo 3d peak of 0.34 eV.

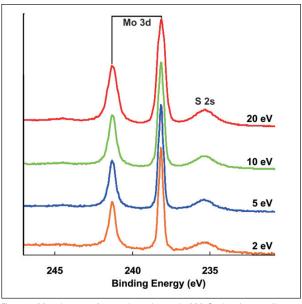


Figure 8: Mo 3d spectra from a cleaved crystal of MoS_2 showing excellent energy resolution

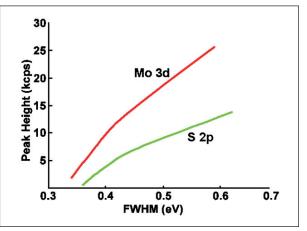


Figure 9: Mo $3d_{5/2}$ and S 2p peak intensities as a function of resolution from a sample of MoS_. The ultimate FWHM is 0.34 eV.

Lens-Defined Small Area XPS

Use of the in-lens apertures, Figure 2 and the XR3 twin anode X-ray source enables XPS analysis of defined small areas of the sample.

The apertures in the lens define both the analysis area and the acceptance angle of the lens. Restricting the range of input angles minimizes tailing of the analysis area, providing accurate analysis of small features.

Figure 10 shows the method used to determine the lateral resolution of the analyzer. A silver knife edge is translated across the analysis area while monitoring the intensity of the Ag 3d peak. The lateral resolution is then defined as the distance through which the sample has to be moved for the signal to change from 20% to 80% of its maximum intensity.

This method of small area XPS is used when the X-ray source delivers a broad beam, e.g. the XR3 twin anode source.

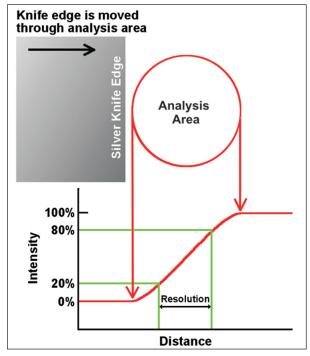


Figure 10: The method for determination of lateral resolution

The signal intensity is, of course, affected by the choice of analysis area. Figure 11 shows the effect of lateral resolution upon the Ag $3d_{5/2}$ peak intensity.

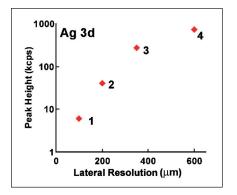


Figure 11: The effect of the lateral resolution upon the Ag $3d_{5/2}$ signal intensity, using Mg K radiation. FWHM = 1.4 eV

Source-Defined Small Area XPS

By using an X-ray monochromator, such as the Thermo Scientific XR5, the analysis area is defined by the X-ray spot size. This method provides higher sensitivity than lens-defined SAXPS. Small spot X-ray monochromators also provide better energy resolution than large spot monochromators.

Source-defined small area analysis has two major advantages over lens-defined SAXPS:

- 1. At a given analysis area, the analyzer's lens can be operated at maximum transmission, providing greater sensitivity.
- 2. X-rays are striking only the part of the sample being analyzed. This means that other parts of the sample or other samples are not subject to X-ray degradation while they await analysis.

Using the XR5 monochromator, the following analysis areas are available.

| POWER (WATTS) | NOMINAL SPOT SIZE (µM) |
|---------------|------------------------|
| 200 | 650 |
| 150 | 500 |
| 100 | 400 |
| 75 | 320 |
| 55 | 200 |
| 45 | 150 |
| 30 | 120 |

Table 1: The spot sizes available from the XR5 and the X-ray power available at each spot size. Other spot sizes within the range 120 to 650 μm can be provided if required.

Figure 12 shows the sensitivity as a function of the peak width for three monochromator spot sizes. These data refer to the Ag $3d_{5/2}$ peak from a clean silver sample. The ultimate resolution for small spot XPS is 0.44 eV, slightly superior to the performance in the large spot mode.

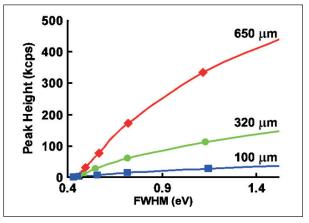


Figure 12: Sensitivity as a function of resolution for three of the available monochromator spot sizes

Angle-Resolved XPS

One of the aperture combinations in the lens provides a $\pm 1^{\circ}$ acceptance angle for photoelectrons. This will allow angle-resolved XPS with high angular resolution.

If used in combination with the XR5 microfocusing monochromator, the analysis area is always larger than the X-ray spot diameter, see Figure 13. This arrangement ensures maximum efficiency for data collection.



Figure 13: ARXPS using the microfocus monochromator

Figure 14 shows the spectrum from silicon dioxide on silicon acquired at two different collection angles, illustrating the relative enhancement of the oxide peak at grazing emission. In addition, this spectrum illustrates the excellent energy resolution available using the Alpha110, the two peaks from elemental silicon being clearly resolved.

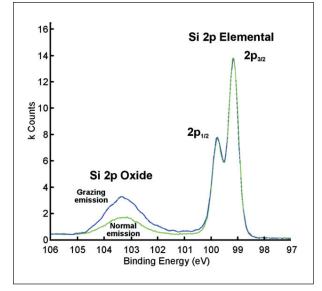


Figure 14: ARXPS of SiO_2 on Si acquired using the microfocus monochromator. Spectra normalized to elemental silicon.

Ultra-violet Photoelectron Spectroscopy (UPS)

The low kinetic energies and the requirement for very high resolution make UPS a very demanding test for any analyzer.

- The Alpha110 is an ideal analyzer for UPS because:The analyzer housing is fabricated from mu-metal resulting in the optimum magnetic shielding. This is especially important when analyzing electrons with very low kinetic energy.
- User-selectable entrance and exit slit sizes control the energy resolution and sensitivity.
- The alpha-plates, shown in Figure 3, ensure very high energy resolution.
- The resolution of the analyzer is partly determined by the minimum step size in a scan. A user controllable step size down to 0.312 meV ensures that the step size does not limit the spectral resolution.

Typically, UPS performance is assessed using the spectrum from the valence band of silver. The intensity of the Ag 4d peak is measured along with the resolution of the Fermi Edge at room temperature. The spectra shown here demonstrate that a peak intensity of 3.2 Mcps can be achieved when the Fermi edge resolution is 87 meV (using the 20% - 80% definition).

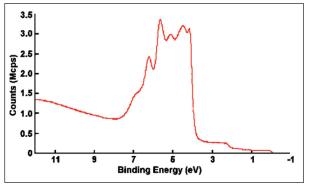


Figure 15: UPS spectrum from silver acquired at room temperature using the Alpha110

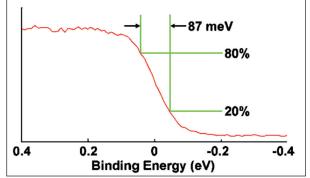


Figure 16: An enlargement of Figure 16 showing a Fermi edge resolution of 87 meV. Data collected using a pass energy of 2 eV.

Auger Electron Spectroscopy

The Alpha110 is the ideal analyzer for Auger electron spectroscopy and scanning Auger mapping because:

- The 7 channel detector provides high sensitivity with an excellent signal to noise ratio
- The analyzer can be operated over a wide range of retard ratios, allowing the optimum resolution and sensitivity to be selected

By using a combination of the Alpha110 and the FEG1000 field emission electron gun, high performance AES and SAM are available.

The FEG1000 produces a spot size of < 95 nm at a current of 5 nA, providing high sensitivity and excellent spatial resolution.

The combination of the analyzer and field emission electron gun also provides fast AES mapping due to:

- The small spot size and high current
- Simultaneous peak and background mapping

Figure 17 shows an example of an AES survey spectrum from copper.

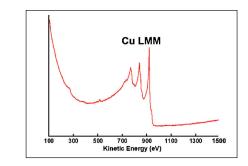


Figure 17: AES survey spectrum from copper

Being a spherical sector analyzer, the Alpha110 can be operated over a range of retard ratios. This allows the user to select the most appropriate compromise between resolution and sensitivity.

Figure 18 shows the effect of varying the retard ratio upon the resolution of the Cu LMM peak. While Figure 19 shows the relationship between peak intensity and resolution for Cu LMM.

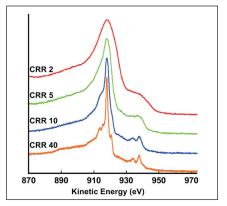


Figure 18: Cu LMM peak acquired at each of four different retard ratios

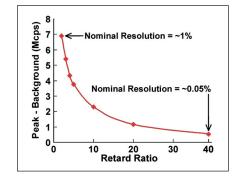


Figure 19: The relationship between peak intensity and resolution for the Cu LMM peak. Data acquired using a 5 keV electron beam at 20 nA.

Note: To obtain the optimum spatial resolution, adequate vibration isolation must be provided.

Ion Scattering Spectroscopy (ISS)

The Alpha110 is equipped with digital, bi-polar electronics. This allows the polarity to be easily switched for ISS studies. The angle resolving lens mode ensures good mass resolution in ISS spectra.

The Thermo Scientific EX05 ion gun is a suitable source for ISS analysis. Figure 20 shows a typical spectrum for a 1keV He⁺ ion beam incident on a gold sample and illustrates the high sensitivity and good resolution achievable.

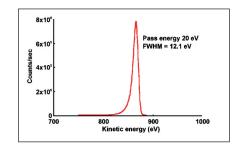


Figure 20: ISS spectrum from a gold sample acquired using He⁺. (Ion energy 1 keV, current 100 nA).

Avantage Data System

Alpha110 is computer controlled, using the same data system featured on the range of high-performance instruments such as Theta Probe, MICROLAB 350 and ESCALAB 250. Use of a common software package across the product range ensures the highest level of commitment, development and support to all *Avantage* users.

Avantage is based on a Windows platform allowing easy integration with report writing software, laboratory management systems and computer networks.

Avantage provides complete control of the operating parameters of the Alpha110, controlling the spectrometer, the lenses and the detector.

In data processing, *Avantage* incorporates all the features necessary to extract the maximum amount of information from the analysis, including:

- Linear, Shirley and Tougaard background subtraction
- Peak identification
- Quantification
- Peak fitting
- Non-linear least squares fitting
- Depth profile construction
- Multi-dimensional data display
- Multiple instrument control

Avantage can also control a range of components, allowing integration of those components with the analyzer, these include:

- Twin anode X-ray source (XPS)
- X-ray monochromator (SAXPS)
- Field emission gun (AES, SEM, SAM)
- EX05 ion gun (depth profiling, ISS, sample cleaning)

Avantage can also be used with sample stages or manipulators to control sample alignment, providing precise sample positioning.

For more information on *Avantage* please refer to the document AN30005.

Specifications

XPS Specifications

To determine the specifications, the sample is placed so that the sample normal is parallel with the lens axis. In addition, the analyzer to X-ray source angle is set at 55°, to eliminate the dependence of peak intensity on the asymmetry effects. This simplifies quantification. (See R.F. Reilman et al., J. Electron Spec. & Rel. Phen. 8 (1976) p389-394.)

For selected area analysis, the sample must be placed normal to the input lens to ensure a uniform, symmetrically shaped projection into the analyzer, see Figure 21.

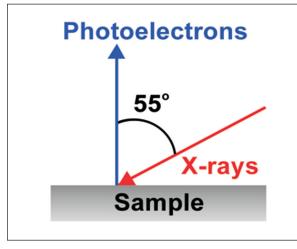


Figure 21: Geometry used to measure the specifications reported here

Large Area XPS

Table 2 shows the specified performance for large area XPS using the geometry illustrated in Figure 22 and a working distance of 23.5 mm.

| FWHM(EV) | PEAK HEIGHT (MCPS) |
|----------|--------------------|
| 0.85 eV | ≥1.0 * |
| 1.40 eV | ≥10 * |

Table 2: Minimum large area XPS performance. Test conditions: 300W Mg K α radiation on Ag3d_{5/2} with background subtracted.

*An input lens angle of 90° would typically produce an increase of around 40% in count rate

Small Area XPS

For the measurement of the performance of the Alpha110 in small area XPS, the analysis area is determined using the method illustrated in Figure 11. Again, the geometry used is that shown in Figure 22, ensuring that the analyzed area is circular.

| ANALYSIS AREA (MM) | PEAK HEIGHT (KCPS) |
|--------------------|--------------------|
| 100 | ≥4.5 |
| 200 | ≥35 |
| 350 | ≥270 |
| 600 | ≥700 |

Table 3: Minimum small area XPS performance. Test conditions: 300W Mg K α radiation on silver with background subtracted at 1.4eV FWHM (Ag3d_{5/2}).

Monochromated XPS

The performance of the Alpha110 with the XR5 microfocus monochromator is shown in Figure 22. These data are obtained using the largest spot size available from the XR5 monochromator.

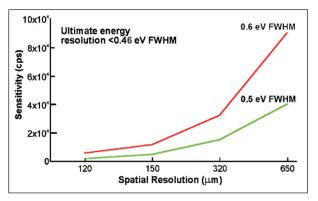


Figure 22: Monochromated XPS performance. Test conditions: XR5 twin crystal monochromator with Al K α anode at various power levels on Ag3d_{5/2} with background subtracted. Sample normal to lens axis.

UPS Specifications

UPS performance is measured using He I radiation from the Thermo Scientific UV-source. The specified minimum performance is shown in Table 4.

| FERMI EDGE RESOLUTION | AG 4D INTENSITY (MCPS) |
|-----------------------|------------------------|
| 90 meV | ≥2.5 |
| 100 meV | ≥4.0 |
| 140 meV | ≥10.0 |

Table 4: UPS performance. Test conditions: He I at 21.2 eV on room temperature Ag sample with background subtracted

AES Performance

Table 5 shows the minimum AES performance that can be achieved using the Alpha110. In this case, the sample is sputter-cleaned copper.

| PRIMARY BEAM | PEAK HEIGHT (MCPS) |
|--------------|--------------------|
| 10 kV, 10 nA | ≥1.5 |
| 5 kV, 10 nA | ≥1.75 |

Table 5: AES performance. Test conditions: Cu LMM at 918 eV with background subtracted.

ISS Performance

The ISS minimum performance is demonstrated using helium ions at 1 keV generated using the EX05 ion gun. A gold sample is used and a linear background is subtracted from the peak height.

| ION CURRENT (NA) | RESOLUTION (FWHM, EV) | PEAK HEIGHT (KCPS) |
|------------------|------------------------------|--------------------|
| 10 | 14 | ≥100 |

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Table 6: ISS specified performance

Dimensions

The Alpha110 can be fitted to a 6" OD mounting flange or a 4.5" OD mounting flange. If a 4.5" mounting flange is used the tube ID must be greater than 65 mm. An FC70 mounting port is recommended. The important dimensions of the Alpha110 are shown in Figure 23.

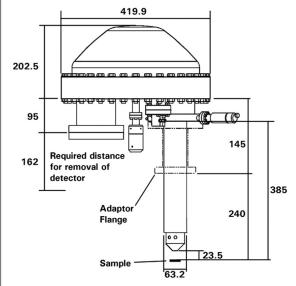


Figure 23: Alpha110 dimensions

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