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KRATOS ANALYTICAL
A SHIMADZU GROUP COMPANY

Superior performance, high throughput imaging X-ray photoelectron spectrometer

AXIS NOVA²

X-RAY PHOTOELECTRON SPECTROSCOPY

X-ray photoelectron spectroscopy (XPS), also known as electron spectroscopy for chemical analysis (ESCA), is a widely used surface analysis technique for materials characterisation. With a standard Al Kα excitation source, the technique provides quantitative elemental and chemical state information from the upper most 10 nm of a material. XPS is used in diverse applications, ranging from defect analysis on microelectronic bond-pads to drug eluting thin films in the pharmaceutical industry.

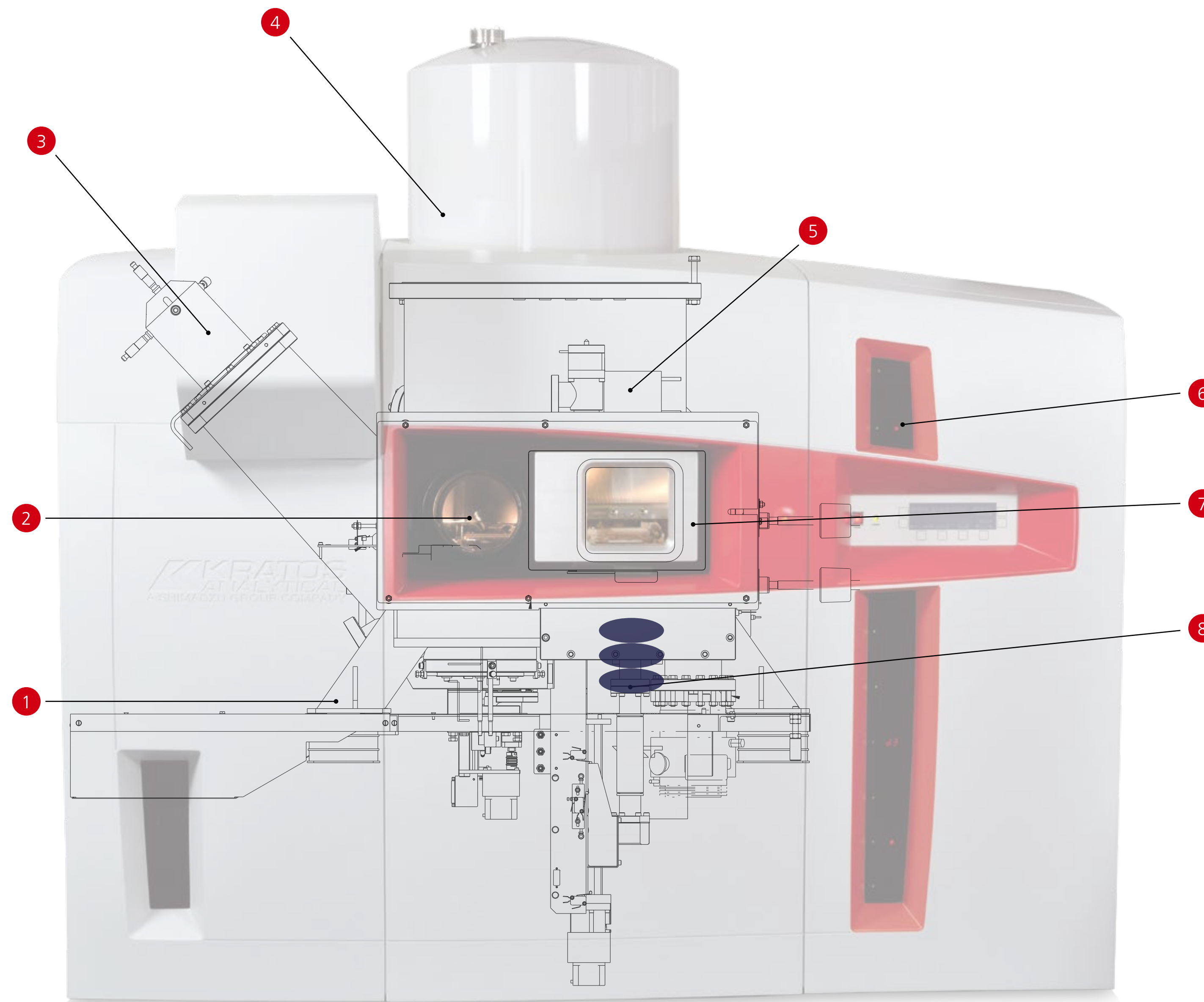
STATE-OF-THE-ART PERFORMANCE

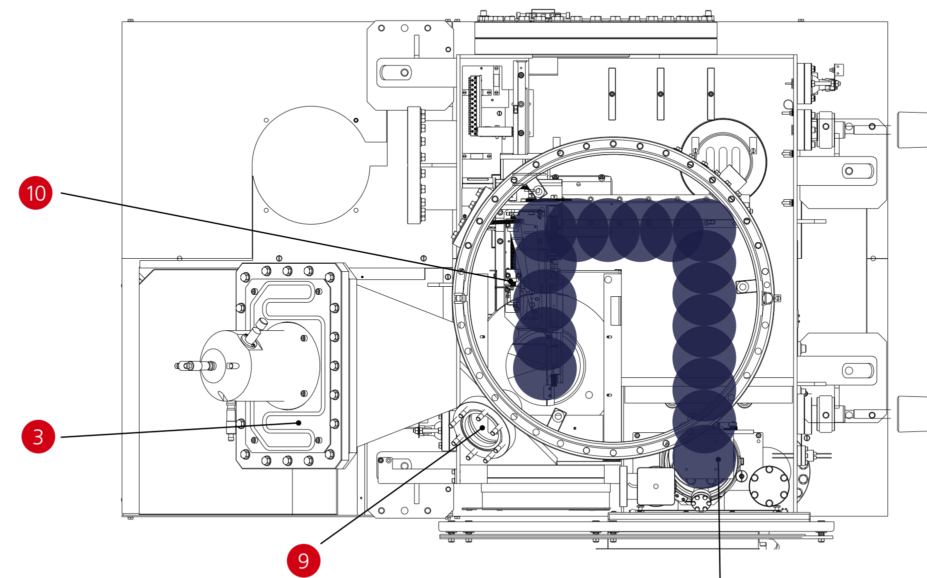
The AXIS Nova² combines XPS imaging and spectroscopic capabilities with a highly automated, large sample handling system and is the next generation of AXIS Nova spectrometer. The AXIS Nova² provides enhanced performance over its predecessor and is based on Kratos' proven AXIS technology comprising: magnetic and electrostatic transfer lenses; co-axial electron-only charge neutralisation; spherical mirror and hemispherical electron energy analysers. Kratos developed innovations such as the delay-line detector for spectroscopy and imaging modes and high energy X-ray excitation sources ensure the AXIS Nova² is capable of performing in the most demanding research and development environments.

Designed for ease of use, the AXIS Nova² has automated sample loading, orthogonal cameras for easy sample positioning and intuitive data acquisition software. A unique capability of the AXIS Nova² is the 110mm diameter sample platen allowing unrivalled large sample handling and high sample throughput. None of these attributes compromise the performance. The AXIS Nova² is capable of high sensitivity, excellent energy resolution and fast, high spatial resolution imaging meeting the analysis needs of the most challenging applications.

COMPACT FOOTPRINT

The AXIS Nova² is designed as a single unit with a compact footprint measuring 2.06 x 1.07m. Additional space is required for a small closed loop water chiller and computer desk. Services required include dry nitrogen vent gas, argon for ion sputtering and compressed air for pneumatic valves all of which are input via a discrete fluids panel at the rear of the spectrometer. The vacuum chamber has integrated heaters and baking covers for easy computer controlled bake-out of the system when required.



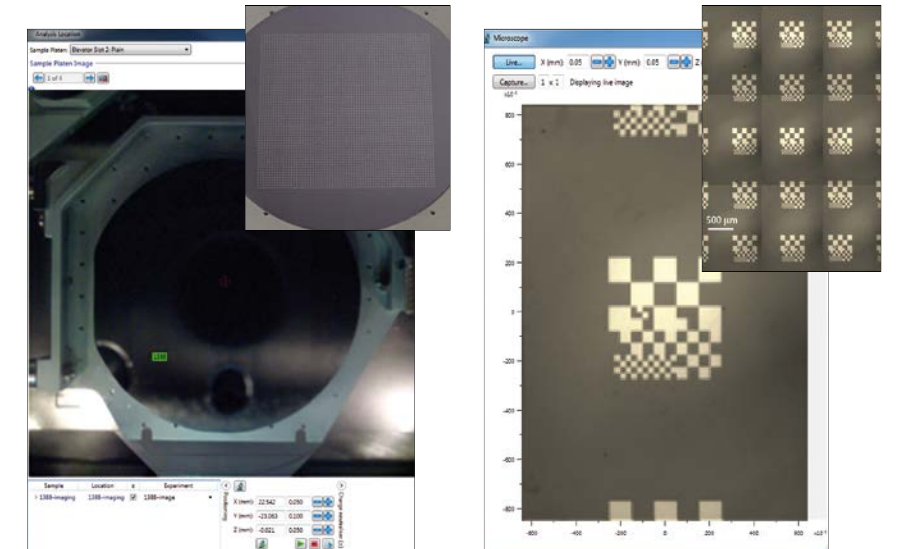


Path of sample platens from the Sample Entry Chamber (SEC) to the Sample Analysis Chamber (SAC). Up to three platens can be stacked vertically in the SEC, on a storage elevator.

1. X-ray source
2. SAC window
3. X-ray mirror
4. SMA and HSA
5. SEC camera
6. Control units
7. SEC window
8. Platen elevator
9. Ion source
10. SAC camera

AUTOMATION TO ENSURE HIGH SAMPLE THROUGHPUT

The sample entry chamber (SEC) accommodates up to three sample platens on a storage elevator. An orthogonal camera in the SEC provides an image of the entire platen allowing the analysis position for samples to be defined. For large, homogeneous samples the sample can be translated directly to the analysis position and data acquisition started. For samples with smaller features or complex structure a high magnification optical microscope in the sample analysis chamber (SAC) can be used to define the analysis region of interest.



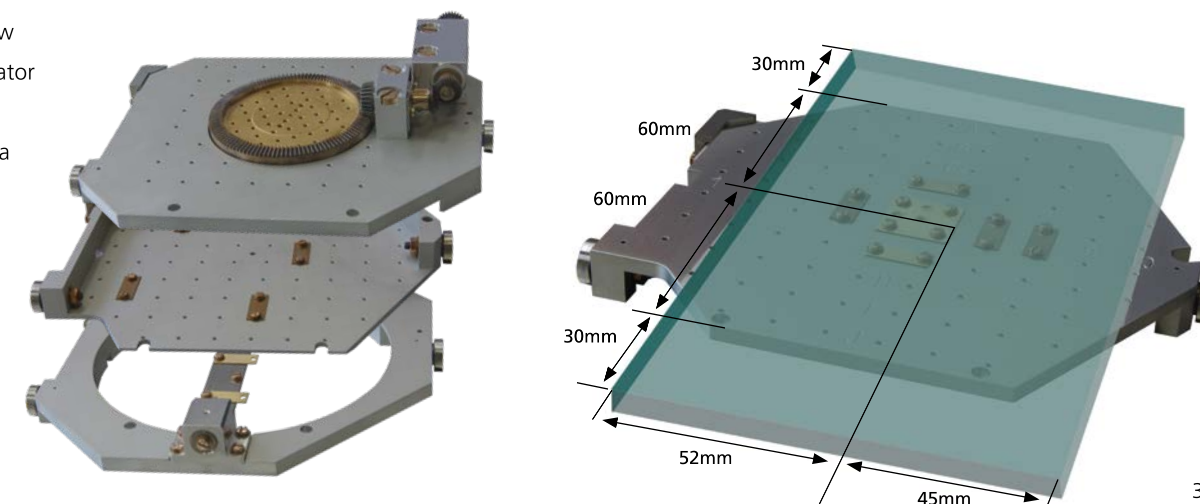
SEC microscope image of the entire platen allowing definition of the analysis position with (inset) digital zoom of the same image to reveal greater detail.

Higher magnification SAC optical microscope image and (inset) 'stitched' 5x5 SAC optical image.

AUTOMATED PLATEN EXCHANGE

Automated multi-platen exchange allows transfer between the sample storage elevator in the SEC and SAC analysis position without User intervention ensuring high sample throughput. Large view ports in both the SEC and SAC mean that the platen can always be observed for complete piece of mind.

To accommodate particular data acquisition requirements platens with specific functionality are supplied. For thicker samples a 'deep sample platen' is used to increase the maximum sample thickness to 19 mm. For thin film angle resolved experiments samples must be mounted on a 'tilt-platen' which allows rotation of the sample about the Y-axis. Rotation during sputter profiling is achieved using the 'azimuthal platen' allowing off axis compucentric rotation about the vertical axis to minimise sample roughening caused by ion bombardment.

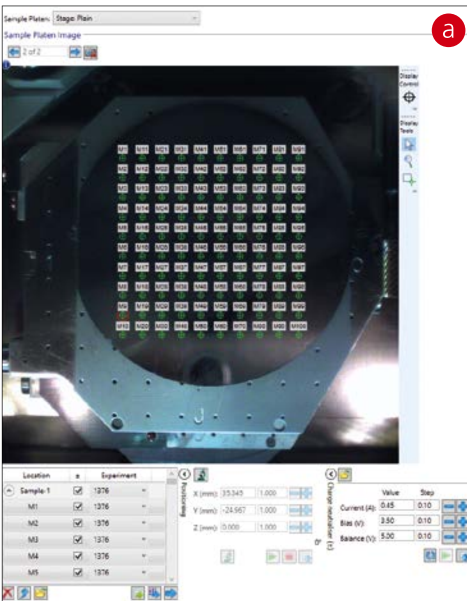


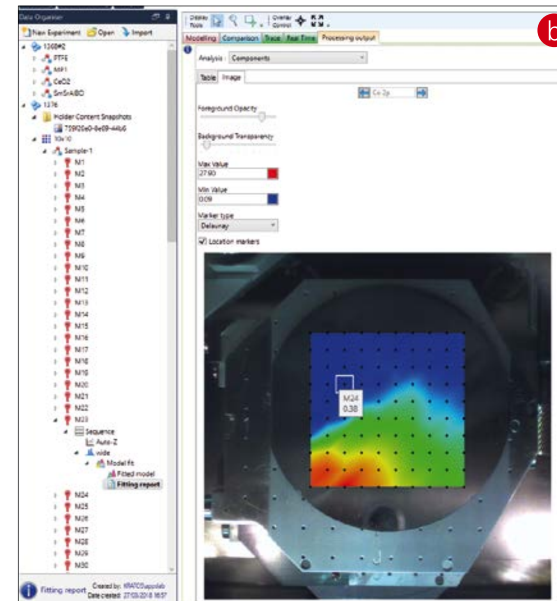
ESCApe SOFTWARE FOR ACQUISITION AND PROCESSING

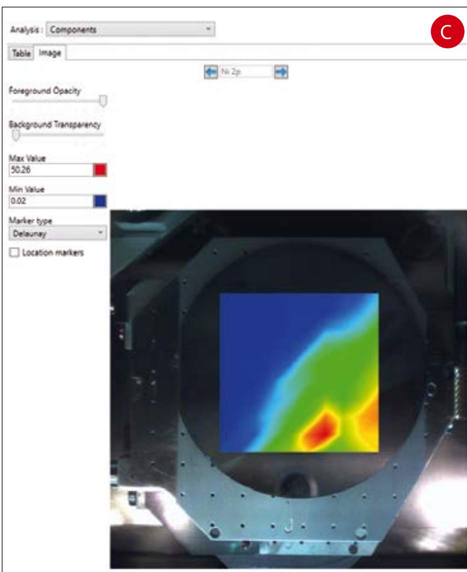
ESCApe software integrates data acquisition and processing allowing the User to exploit the benefit of this highly automated spectrometer. Following identification of the analysis position from the optical microscopes, predefined acquisition methods are selected to start sample analysis. These methods can be as simple as a survey spectrum or more complex such as sputter depth profiling with compucentric rotation during etching. There is flexibility for expert Users to define their own acquisition methods.

Acquisition methods associated with samples on any of the three samples platens can be added to the analysis queue. Where analysis is defined from samples on different sample platens they are exchanged as part of the automated work flow, exploiting the high throughput capability of the spectrometer. For large sample analysis an array of analysis positions can be easily defined within the ESCApe software. Processing is no longer a rate-limiting step with automated peak identification and quantification integrated into the ESCApe software.

For more information, click here.



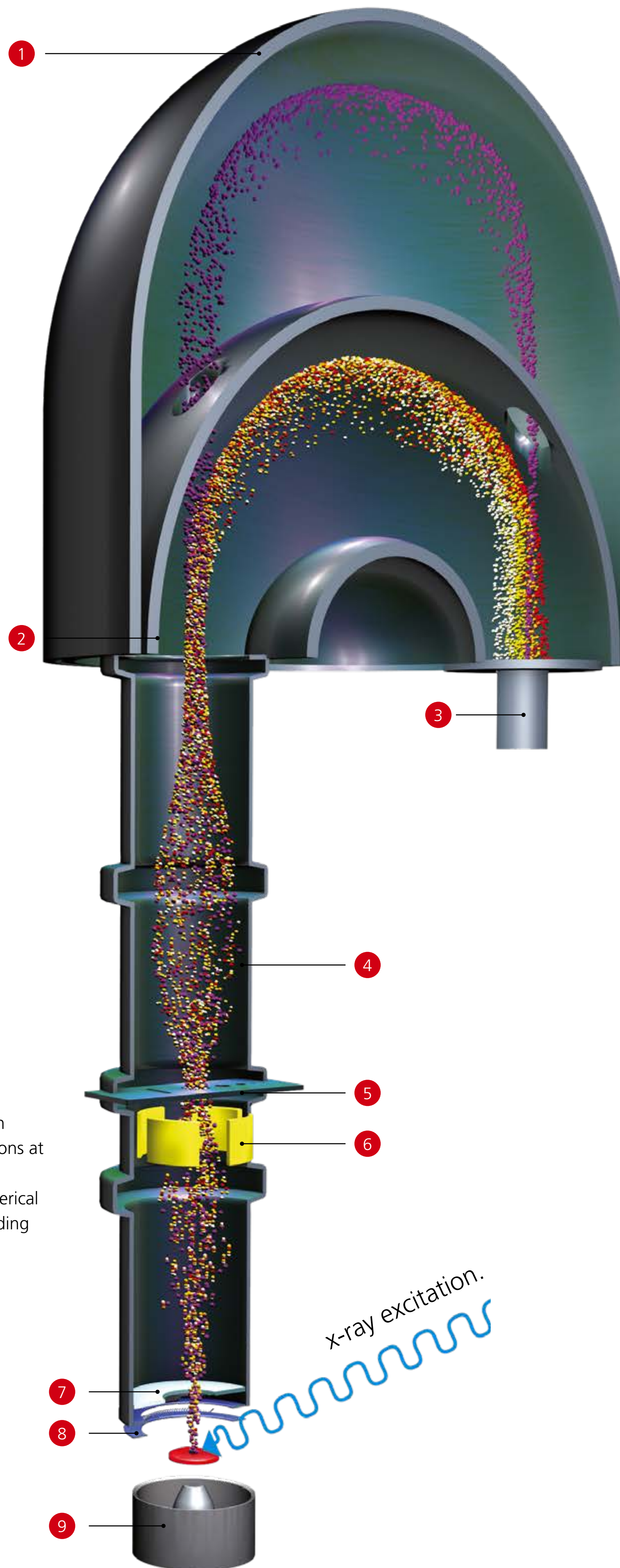




'Array analysis' from a 3-target combinatorial sputter coating of a 4-inch wafer. (a) ESCApe allows easy definintion of the analysis array across the wafer. (b) data can be displayed as a colour concentration plot overlaid on the optical image of the wafer with simple mouse click to link to each individual spectrum. Image (c) shows the Ni colour concentration plot complimentary to the Cr plot shown in (b).

The patented AXIS technologies of magnetic and electrostatic lenses ensure high electron collection efficiency in spectroscopy mode and low aberrations at high magnifications in parallel imaging mode. The unique combined high transmission hemispherical and spherical mirror analysers guarantee outstanding spectroscopic and parallel imaging performance.

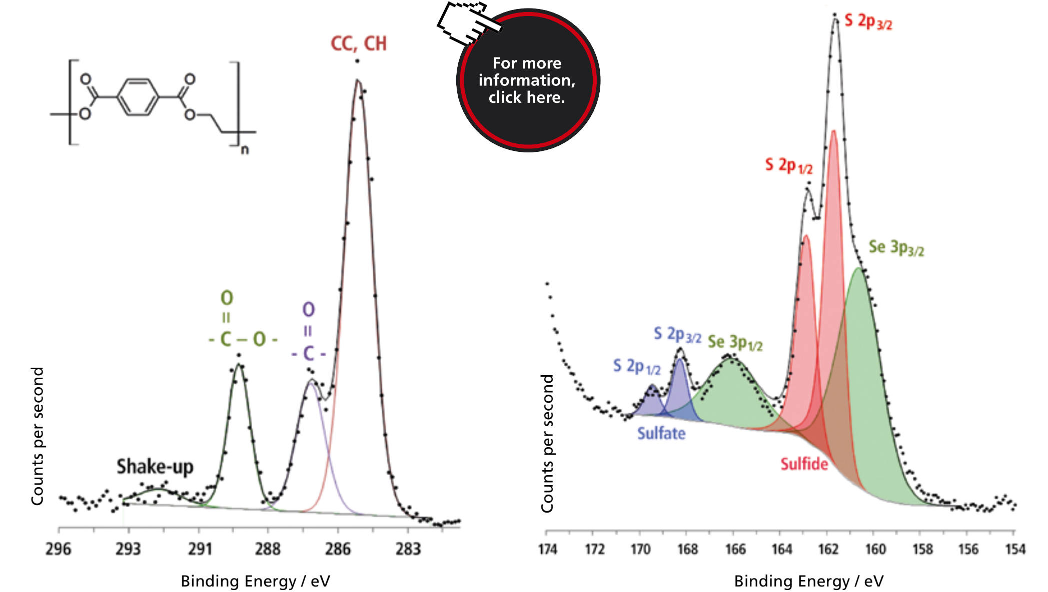
1. Spherical mirror analyser
2. Hemispherical analyser
3. Delay-line detector
4. Electrostatic lens
5. Selected area aperture drive
6. Octopole scan plates
7. Variable iris drive
8. Charge neutraliser
9. Magnetic lens



LARGE AREA, HIGH SENSITIVITY

An important aspect of any XPS instrument is spectroscopic performance. The AXIS Nova² is designed for the very best performance at large analysis area with efficient collection of photoelectrons contributing to high sensitivity. The spectrometer achieves unrivalled sensitivity by using both magnetic and electrostatic lenses in combination with the large 165 mm mean radius hemispherical analyser. Photoelectrons are counted using a multichannel plate stack above a delay-line detector with 128 data channels. This detector provides the capability of acquiring spectra in unscanned snapshot mode where spectra are collected without scanning the lens/analyser. This mode of acquisition has the significant advantage that a core level spectrum can be collected in less than a second.

For more information, click here.



C 1s unscanned, snapshot spectrum from PET polymer acquired from 55 µm in 30 s.

High resolution spectrum of the S 2p & Se 3d region of a sulfur contaminated CdSe sample acquired in large area, high sensitivity mode.

The large, 500 mm Rowland circle, X-ray monochromator produces high energy resolution spectra which may be collected in a matter of seconds. A moveable, position-indexed, X-ray anode means that the useful lifetime of the anode is significantly extended. An optional dual anode monochromatic X-ray source, combing Al Kα and higher energy Ag Lα, can be specified. The advantage of the Ag Lα (2984.2 eV) X-ray source is that the information depth can be extended to ca. 15–20 nm and additional, higher binding energy, core levels can be excited.

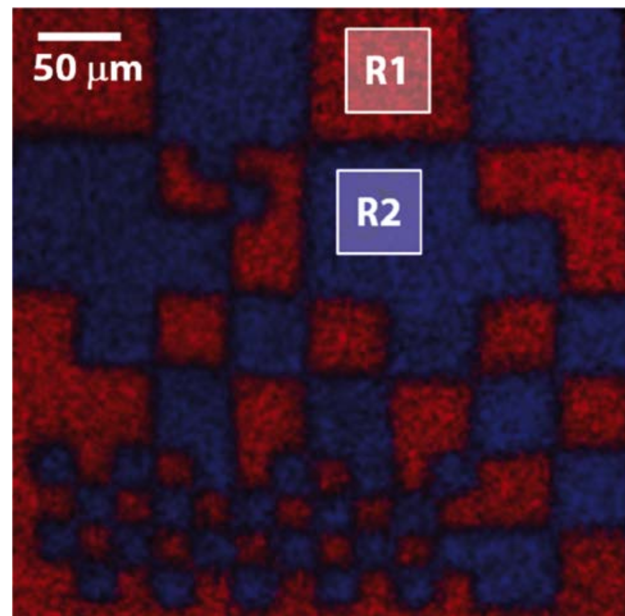
Outstanding performance is not limited to conducting samples. Use of a low energy, electron only charge neutralisation system allows photoelectron spectra to be collected from insulating, topographic samples with ease.

THE AXIS NOVA²: MADE TO MEASURE

HIGH SPATIAL RESOLUTION XPS IMAGING

As well as impressive spectroscopic performance, the AXIS Nova² incorporates a spherical mirror analyser (SMA) for high spatial resolution XPS imaging. In parallel imaging mode the spatial distribution of the photoelectrons is retained as they are projected onto the 2-dimensional delay-line detector. Stigmatic, parallel images can be collected in a matter of seconds offering shorter acquisition times and higher spatial resolution than sequential rastered beam or stage mapping approaches.
An attribute of the SMA is that it operates in fixed analyser transmission mode ensuring that the energy resolution of photoelectron images is constant for all kinetic energies. This is of particular importance for quantitative imaging applications. Parallel images may also be acquired at lower pass energies to improve the energy resolution, analogous to spectroscopy mode, allowing chemical state imaging where necessary.

The low spherical aberration of the electron optics ensures that the image of the surface can be magnified onto the detector with very little distortion. This ensures high spatial resolution images. Parallel imaging at the highest magnification gives a guaranteed spatial resolution of 3 µm.



Spectromicroscopy data acquired at 80 eV pass energy through the spherical mirror analyser of Al pattern on Si wafer. The image shows the Al (blue) and Si (red) over the 400 µm field of view. Spectra, (right) demonstrating the excellent energy resolution, were reconstructed by summing the pixels as a function of energy from the regions indicated on the image.

SPECTROMICROSCOPY

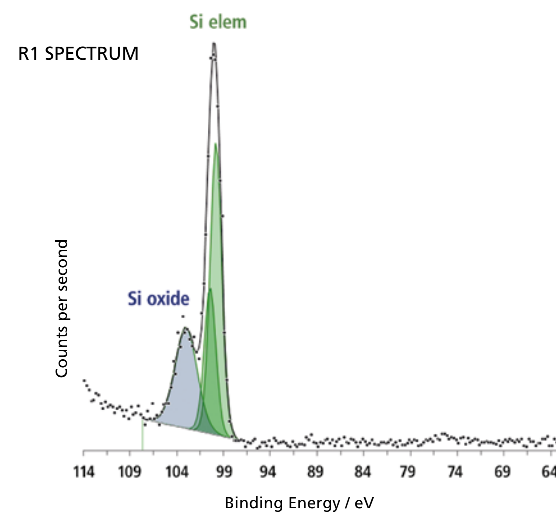
A series of fast images can be acquired as a function of energy allowing the extraction of spectra from images (spectromicroscopy data with over 65,500 spectra over the defined field of view). Such datasets are ideally suited to multivariate analysis which can be used to partition the data from noise and reconstruct spectra form single pixels. After extracting the spectra, components may be fitted using conventional peak fitting approach with the significant advantage that the components may then be used to reconstruct quantitative chemical state images that would not be possible in conventional parallel imaging XPS.

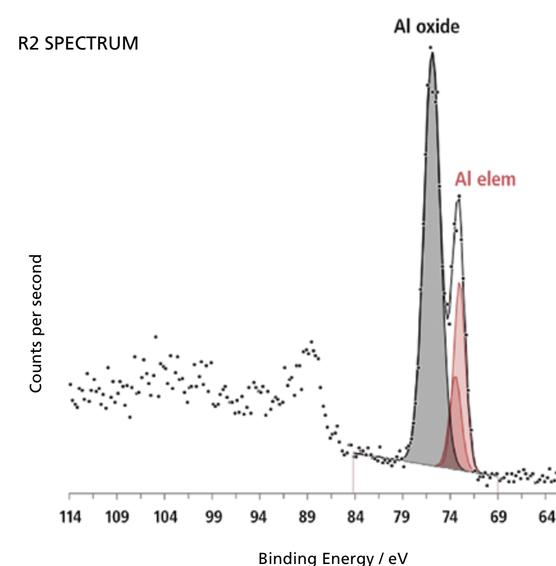
MULTI-TECHNIQUE CAPABILITIES

Whilst the AXIS Nova² is primarily designed for high throughput high performance XPS additional analytical capabilities can be added without compromising the performance. An ultraviolet He-discharge lamp can be added to allow collection of ultraviolet photoemission spectra (UPS) for valence band and work function measurements.

Ion scattering spectroscopy (ISS) can be configured as an additional technique for elemental characterisation of the outermost surface of the sample. Both the Minibeam 4 and 6 ion sources can be configured for use with low energy He^+ ions and the analyser polarity reversed to achieve this acquisition mode simply through the ESCApe User interface.

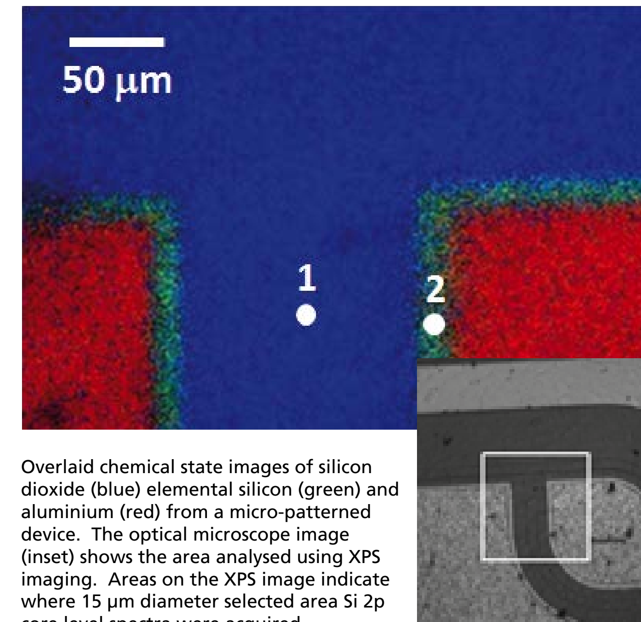
For more information, click here.



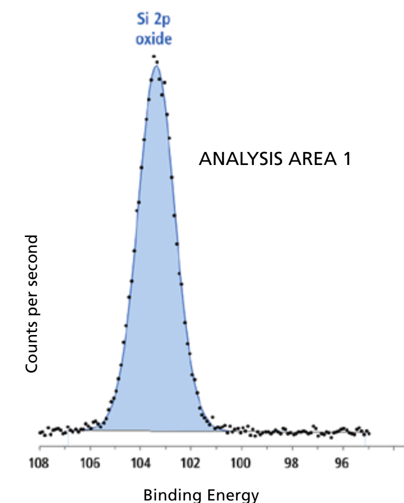


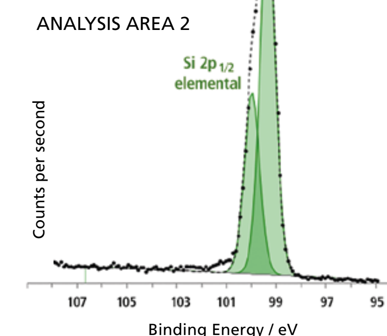
XPS FROM SELECTED AREAS

Selected area spectroscopy is performed by inserting an aperture into the electrostatic lens column, forming a virtual probe at the surface of the sample. Spectra are acquired from defined selected areas down to 15 µm diameter with improved performance over the first generation instrument. Multipoint spectra can be acquired from any position within the field of view using an electrostatic deflection system. A simple mouse click is used to define the analysis position from an image deflecting the virtual probe using electrostatic scan plates. This approach removes the need to move the sample and hence uncertainties introduced with repeated sample translation.



Overlaid chemical state images of silicon dioxide (blue) elemental silicon (green) and aluminium (red) from a micro-patterned device. The optical microscope image (inset) shows the area analysed using XPS imaging. Areas on the XPS image indicate where 15 µm diameter selected area Si 2p core level spectra were acquired.





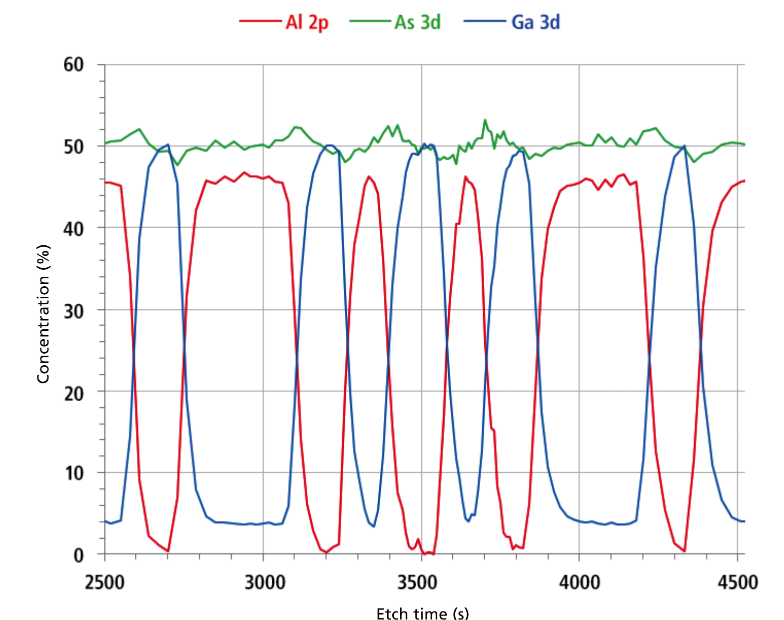
ION SPUTTER DEPTH PROFILING

The AXIS Nova² can be configured with a standard floating column monoatomic Ar^+ ion source or the Gas Cluster Ion Source (GCIS) depending on the type of sample to be profiled. With either ion source ESCApe software allows compucentric rotation during sputtering to minimise ion induced sample roughening during etching.

The standard monatomic Ar^+ ion source **(Minibeam 4)** provides continuously variable beam energies between 4 keV and 50 eV. The ion column has a bend to suppress energetic neutrals as well as the capability to operate in floating mode for high ion beam current density at low ion energies. Float mode gives the advantage of improved interface resolution and fast etch rates even at low ion acceleration voltage.

The multi-mode GCIS **(Minibeam 6)** is capable of generating Ar_n^+ clusters consisting of hundreds or even thousands of Ar atoms as well as monatomic Ar^+ ions for depth profiling and He^+ for optional ion scattering spectrometry (ISS). The use of cluster ions allows the successful depth profiling of 'soft' organic polymers with retention of chemistry. The energy per projectile atom, or partition energy, can be as low as a few electron volts. It has been shown that these cluster ions sputter material from the near-surface, causing very little sub-surface damage so that excellent interface resolution can be maintained through multilayer depth profiles of several microns. Ar_n^+ cluster mode depth profiling is also finding application to inorganic depth profiling where small (n=500) clusters accelerated to 20 keV cause less preferential sputtering than conventional monatomic Ar^+ ions when etching inorganic materials.

Pre-defined ion source operating conditions provided in a look-up table allow easy use of either ion source. Automation extends to the argon gas supply for the ion source which can be turned on and off as required during unattended operation, pressure in monatomic mode being controlled by an automatically regulated piezoelectric valve.



4 kV Ar^+ depth profile of alternating layers GaAs / $Al_xGa_{1-x}As$, a model single photon LED device, expanded to show the emitter region corresponding to layers 15 – 19.

SYSTEM SUMMARY

Vacuum System

Sample Analysis Chamber

- 400 l/sec Turbomolecular pump with backing rotary pump. Auxiliary titanium sublimation pumping.

Sample entry chamber

- Flexible sample load lock, Sample elevator accommodates up to 3 sample platens, Turbomolecular pump (220 l/sec) with rotary pump backing.

Excitation Sources

High power monochromatic X-ray source

- Multi-position Al (standard) or Al/Ag anode (option)
- 500 mm Rowland circle
- Single quartz toroidal backplane
- Computer control with read-back and interlocks
- Ultra Violet lamp He I / He II discharge (option)

Electron Energy Analysers

- 180° hemispherical analyser (spectroscopy)
- Spherical mirror analyser (parallel imaging)

Photoelectron Detector

- Multichannel plate array with delay-line detector
- Scanned & snapshot spectroscopic acquisition
- 2D parallel imaging

Charge Neutralisation

- Co-axial electron only

Sample Mounting & Handling

- Standard sample platen
- Deep sample platen
- Angle resolved platen (0 - 85° sample rotation)
- Compucentric rotation platen (for rotation during profiling)

Software

- ESCApe integrated acquisition and processing software for automated acquisition and instrument control.

Kratos Analytical, a wholly owned subsidiary of Shimadzu Corporation of Kyoto, Japan, has been manufacturing surface analysis instruments since 1969. Throughout this period Kratos has continued to lead the development of new technologies related to X-ray photoelectron spectrometers and associated accessories for surface and materials characterisation.

All Kratos products are designed, assembled and supported from our headquarters in Manchester, UK.

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