Argus CU

Next Generation Hemispherical Analyser



- Compression Lens
- Excellent Sensitivity
- True Counting Multi-Anode Detector
- Linear Response up to the Highest Count Rates
- Excellent Dynamic Range
- Snapshot and Dynamic XPS
- Chemical State Mapping



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ESCA⁺ system with the new Argus CU hemispherical analyser.









One Step BEYOND Conventional ESCA

Argus is the next generation hemispherical analyser with multi-channel detection technology developed and optimised for uncompromised photoelectron spectroscopy.

Argus represents a new and unique approach to multichannel XPS detection, matching today's demanding requirements for fast and trusted quantitative XPS, ease-of-use and reliability.

The Argus hemispherical analyser has superior sensitivity, new detection modes and allows for faster sample analysis data acquisition rates with

when compared to channeltron based systems. A multi-anode detector inside the Argus employs 128 individual counters connected to a striped-anode array (see Fig. 1).

As a key technology, dedicated low-noise in-vacuum pulse counters with high detection efficiency provide for optimum a theoretical maximum readout significantly larger than 100 Mcps integral count rate. The Argus design enables extremely fast snapshot acquisition of spectra with minimised crosstalk between neighbouring channels. The detector's linear response up to the highest count rates is a prerequisite for trusted quantitative analysis.

The optimised and well adapted imaging lens system in combination with the 128 channel detector allows analysing the sample with ultimate accuracy.

The Argus CU Concept

Compression Lens meets Multi-Channel Detection · a perfect match.

The new Argus lens design 'Argus CU' incorporates a thoroughly optimized compression unit which delivers a line focus at the entrance of the hemisphere. Unlike conventional axially symmetric lenses, the compression lens technology allows to transform a divergent electron beam originating from an extended round analysis area into a flat beam that passes the entrance slit without transmission losses. This lens design is therefore highly advantageous over conventional analyser lenses for the following reasons.

Argus CU includes a unique 128 channel stripe anode detector which provides massive parallel detection. Detector channels at the exit of the

ting through the MCP stack. The amplified electron signals are collected by a stripe anode array which is connected to 128 dedicated amplifiers and counters. Dedicated in-vacuum readout electronics ensure fast data transfer to the ex-vacuum electronics.

hemisphere are orientated along the dispersive plane so that they record 128 different energies in parallel while integrating signals along the non-dispersive plane.

Despite of being a dispersive element the analyzer's hemisphere - in a simplified picture - can be reduced to a non-magnifying lens. This means the image in the entrance is projected 1:1 to the exit of the hemisphere. For multi-channel detectors it is therefore ideal to fill the hemisphere's entrance with a line shaped electron beam which has similar dimensions as single anode stripes of the multi-channel detector.

The Argus CU fulfils this demand by compressing electrons in the non dispersive plane (see Fig. 1) while keeping transmission high.

As a result the analyser offers outstanding sensitivity, simplified handling and maximum convenience during operation. In particular excellent snapshot performance is obtained by combining large entrance apertures and high pass energies leading to very high count rates maintaining sufficient energy resolution. In addition the Argus CU is accurately characterised for quantification.



Faster Analysis

Argus CU ensures fast sample analysis with superior sensitivity compared to previous **Channeltron detection** systems.

Besides Argus CU's highest sensitivity the key to modern sample analysis are dedicated detection modes such as snapshot XPS, high speed depth profiling and rapid chemical state imaging. Argus CU in combination with the new

XPS control system Matrix provides the user with a new and straight-forward approach to data acquisition helping to maximise the efficiency and experimental throughput.

In addition the state-of-theart-detection system opens up the route to a wide range of exciting new electron spectroscopy experiments such as real time observation of dynamic processes (dynamic XPS) - simply not accessible with earlier conventional detector technologies.

In snapshot XPS extremely high countrates maybe experienced by a small number of detector channels around the peak energy when the data is being collected. Other snapshot detectors e.g. based on delay lines or resistive anodes need careful calibration procedures to handle this. However, various non-linear detector artefacts are difficult to overcome. In contrast, the Argus CU detection system ensures that each detector channel has a well defined position in energy at the analyser exit and is hard-wired to its own amplifier and counter. As a result, signature free true parallel detection is the key advantage of the Argus CU. Simply a robust, versatile, easy to use and fully software integrated XPS detector. Argus CU ensures sample analysis with optimised electron optics and significantly improved count rates.



Scanning & Snapshot XPS

Hemispherical analysers offering the snapshot mode allow for much faster data acquisition compared to conventional serial scanning analysers with channeltron detection.

Multi channel detectors with a sufficient number of channels can generate spectra in snapshot mode with excellent energy resolution. In this mode

each channel records only one energy interval for a given time. Needless to say, that a well chosen number of channels is needed to run the spectrometer in the snapshot mode; the number of channels is a balance between count rate per channel and smallest useful energy increment between each channel at the analyser exit. A perfect match has been chosen to guarantee ultimate snapshot performance with Argus CU.

The snapshot mode is commonly used instead of narrow scans



on individual core level peaks (see Fig. 3 as an example). In addition to a well defined transmission function of the analyser the linearity and homogeneity of the detector is most crucial for trusted quantitative sample analysis.

The measurement in Fig. 3 shows that quantitative analysis with the Argus CU is fully reliable in snapshot mode. Both the scanned and the snapshot spectra provide identical values for quantitative analysis (see table). Compared with scanning, snapshot mode can significantly reduce the time taken for complex samples, multiple samples, depth profiling measurements and chemical mapping.





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Cd 3d5/2 Peak



Fig. 4 A: Light dependent resistor before mounting. Fig. 4 B: See also page 9, Fig. 12. The sample structure can be easily identified when compared with the LDR image (4 A). Fig. 4 C: Fast survey spectrum showing the compounds of the LDR (Cd, S, In, Sn, O and C).



The snapshot series with a time resolution in the ms regime allowed examination the different response times of the LDR. The Cd $3d_{5/2}$ peak shifted slowly to lower kinetic energy with a time constant of $\tau_1 = 133\pm8$ ms after the light was switched off. In contrast to that, the LDR switched back faster to its illuminated conductive condition with a time constant of $\tau_2 = 27\pm4$ ms.



Discover the Dynamics in XPS

Driven by technical advances in analysers and detectors, dynamic XPS has recently become a powerful analysis tool. Many experiments in this field have previously not been accessible with standard detector technology.

Key to this technique is the combination of a fast variation of measurement parameters (e.g. sputter time, temperature and light) and an optimised analyser and detector set-up allowing rapid data acquisition. In detail the detector must provide highest sensitivity, optimum dynamic range and excellent homogeneity even at highest count rates to allow detailed understanding of the underlying processes. Two examples of dynamic XPS demonstrate the capabilities of Argus CU.

The first experiment (Fig. 4,5) was carried out on a standard, commercially available light dependent resistor (LDR) shown in Fig. 4A.

Chemical information was provided by area integral- and imaging XPS. In addition to chemical and lateral information a series of XPS snapshots where recorded on the Cd 3d_{5/2} peak. The snapshot data reveals the dynamic reaction of the LDR to the gated LED illumination, a result that could not be obtained with scanned data acquisition methods.

A second dynamic XPS experiment was recorded highlighting the snapshot capabilities of the Argus CU (Fig. 6-8). Here an energy window (15eV), set to the Si 2p core level peak, was acquired over time on a SiO₂ sample with a high repetition rate and good energy resolution. The snapshot mode is thus ideally suited for real-time XPS observations of fast processes.

Solid State UPS



Fig. 6 & 7: The measurement shows the evolution of XPS spectra while removing an in-situ grown SiO₂ layer on a Si substrate by ramping the temperature from 600 °C to 950 °C within one hour. More than 6000 snapshot spectra have been recorded with an acquisition time of 0.5 seconds per spectrum to follow the time evolution of the subcomponents Si⁴⁺ to Si⁰ during the heating process of the Silicon. The complete removal of the oxide takes place within a very narrow temperature window around 850 °C.

Fig. 8: The removal of the oxide occurred over 2.5 min. - an acquisition time during which 300 spectra have been recorded.

Fig. 9: UPS spectrum on polycrystalline Silver at room temperature excited with the high brightness VUV source HIS 13 operated at He I (hv = 21.2 eV).

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Complex Chemical State Mapping

The Argus CU input lens design includes proven scan deflector technology, allowing effortless, fast and reliable chemical state mapping of the sample.

XPS maps provide full peak information including chemical shifts at each pixel (see fig. 11, 12 & 13).

Instead of serially scanned plain peak (or peak-background) count rate detection (e.g. on the Cd $3d_{5/2}$ peak) the new multi-channel detector records a complete energy interval of the spectrum containing the XPS peak, tails and background at each pixel (see fig. 10). This information is acquired in a short time with lateral resolution down to <60 µm defined by

the analyser's aperture. Straight forward off-line data analysis of such spectral maps allows the understanding of the complex local chemistry of the analyzed sample. Technically the actual mapping is done by the deflectors which scan the analysis area of the analyser across the sample while the sample and X-ray source are kept at a fixed position.

In addition, the deflector technology can also be used to undertake local spectroscopy on multiple regions of the sample without me-

chanical movement of the sample stage. In contrast to other spectrometers used for imaging XPS, the Argus CU electron lens system makes use of a dedicated aperture mechanism minimising tailing of the analysis area. Therefore accurate analysis of small sample features and well-defined image resolution becomes possible. As a result, the Argus CU provides the user with an extremely efficient and straightfoward platform to investigate

complex samples by chemical

state mapping.



The Light Dependent Resistor (LDR) is a good example for today's demand for accurate understanding of samples with complex local chemistry. In the present study the Argus CU was used to reveal the local sample information by chemical state mapping and local spectroscopy.

Fig. 11) to 14) show 4 maps, each consisting of a RGB colour overlay of different elements (11 & 12) and chemical states (13 & 14). Each map includes 40.000 snapshot spectra on each of the 8 different elements (Cd, In, Ag, S, Sn, Ni, C and O). Fig 8) shows raw data of a snapshot on the Cd peak.

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The individual spectra, (fig. 11 A to 11 C) confirm the results extracted by mapping. As an example the green areas in Fig. 11-13 represents a region covered with ITO. The spectra were recorded by point clicking in the marked areas 1-3 (Fig. 11) using MATRIX to record the individual spectra without sample movement. Each small spot spectrum was recorded from a 170 µm area.

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W/ Software/

MATRIX for Electron Spectroscopy

The MATRIX concept for spectroscopy gives a user friendly yet extremely powerful interface for experiment control and data acquisition.

In addition MATRIX is the first commercial platform to offer combined SPM and electron spectroscopy experiments within the same software. All experiments will provide similar handling and workflows – a key for everyday life in a modern laboratory. The spectroscopy user interface immediately allows even inexperienced users to run the full range of experimental techniques through the intuitive control panel.

A well organized region list defines the elemental transitions, pass energy and other parameters to provide optimum control of ongoing experiments. A periodic table of the elements including all relevant element transitions allows the running of predefined scans on selected transitions. A dedicated data browser 'Vernissage' allows simple navigation through recorded data allowing users to maintain an excellent overview of large data sets by using a similar approach to data handling as it is used for example in music data bases. This includes the ability to search for dates, comments and other parameters and also includes 'star'-ratings for individual data sets.

15A

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An example for the powerful software is the access to a wide range of parameters by the MATRIX Automated Task Environment (MATE). MATE allows advanced users to modify and expand the capabilities of the analyser for dedicated purposes far beyond standard acquisition schemes, as it is required for example in synchrotron environments. The combination of MATE with a remote access library allows controlling the analyser remotely by third party programs e.g. LabVIEW, RSI IDL, C/C++, Matlab and many others.

MATRIX features a region list that provides a straightforward overview of the most relevant parameters (15 A). A table of the elements offers easy setup of the planned experiments by predefined regions (15 B). In addition the scan window offers quick access to the data and various analysis tools (15 C).

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15C



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Technical Information

Acquisition modes

- Scanned
- Snapshot

XPS

Intensity vs FWHM. Intensities and FWHM for Ag $3d_{_{5/2}}$ photo electrons excited by 300 W Mg Ka (DAR 450), in high magnification mode.

| Peak-width FWHM (eV) | Countrate* |
|----------------------|------------|
| 0.85 | 8 Mcps |
| 1.00 | 30.0 Mcps |

Monochromated XPS

Intensity vs FWHM. Intensities and FWHM for Ag $3d_{5/2}$ photo electrons excited by Omicron XM 1200 Al Ka, in high magnification mode.

| Peak-width FWHM (eV) | Countrate* |
|----------------------|------------|
| 0.60 | 2 Mcps |
| 1.00 | 6 Mcps |

UPS on solids

Intensity and FWHM. Fermi edge width and Ag 4d peak count rate excited by a HIS 13 VUV source (He I).

| Resolution 16-84% [meV] | Countrate |
|-------------------------|-----------|
| 105 | 20 Mcps |

* 90° angle between Argus CU analyser and x-ray source.

