NEWSFLYER SPRING 2023

DEAR VALUED CUSTOMERS, PARTNERS AND COLLABORATORS!

We welcome you to the 2023 Spring Newsflyer from us at Scienta Omicron.

This year we have a lot to celebrate! Around the year-end of 2022 we reached a milestone in Scienta Omicron´s history; we shipped our 1000th system (and we don´t include bolt-on SPM microscopes, or analysers in that number!). We are humbled by the thought that our instruments, placed all over the world, have now reached this respectable number. We hope to ship many more of our innovative systems and continue to support future researchers´ goals.

Scienta Omicron was founded in 2015 after merging VG Scienta and Omicron NanoTechnology, two of the most renowned companies in the field of Surface Science. VG Scienta was founded in 1983 by Kai Siegbahn, after receiving his Nobel Prize in 1981. Omicron Nanotechnology was founded the following year in 1984. This means that the Scienta part of our company has spent 40 years in the industry this year. That is 40 years of innovation, product development and industry leadership. This is something that we want to celebrate with you! During the year we will look back onto some of our achievements and reminisce in memories created together with all of you. We look forward to, at least, another 40 years in the industry and to keep our tradition of pioneering and leading Surface Science and Nanotechnology innovation.

We also want to take the opportunity thank you, our customers, partners, and collaborators, for your continued and unrelenting support during Scienta Omicron's journey. We are proud to be part of the international research community and look forward to continuing to support your research with our innovative instruments.

Yours sincerely,

Scienta Omicron Team

Earlier this year the Scienta Omicron Team shipped the very first DFS-30, one of the 1 000th shipments we have made over the years. Looking forward to another 1 000 shipments!

In this edition of the Newsflyer you can read about:

- Our newest addition for POLAR SPM Lab with the 1 K pot for experiments at below 1.7 K
- Enhanced ARPES experiments with PEAK at Stanford Synchrotron Radiation Lightsource in the US
- PEAK Slit Control Upgrade for our electron analysers enabling optimal settings with remote control
- A feature about the HiPP Lab and advanced APXPS measurements
- An article about the ongoing partnership between Scienta Omicron GmbH and FOCUS GmbH: Highlights from the new NanoESCA MARIS system
- Installation of an APPES/XPS MIP system at the National Institute for Material Science in Japan

POLAR 1 K

1 K pot NOW available for POLAR SPM

The low temperature POLAR bath cryostat SPM was designed for STM and QPlus® measurements with the highest resolution. The cryostat, which was developed in-house, enables a minimum temperature of T < 5 K with an ultra-low helium consumption, providing an exceptionally long helium hold-time.

POLAR SPM was designed, right from the beginning, to enable the introduction of a number of variants over time. Since the product launch, we have successfully integrated a superconducting magnet for vertical fields of 5 Tesla, a temperature variation facility for fast temperature variations of the sample (e.g. 50 K steps within only 5 minutes!), and the integration of a motorised lens for TERS measurements.

Now we have achieved the next milestone: The integration of a 1 K pot into the very compact bath cryostat of the POLAR. With this option, temperatures of below 1.7 K (typically < 1.5 K) can be realised. Conventional 1 K pots are operated in the so-called “continuous mode”, in which a needle-valve is used to adjust a permanent helium flow through a small 1 K pot volume. With this conventional approach, the flow changes over time, requiring frequent adjustments of the needle valve to keep the flow and temperature constant. In contrast to the conventional design, in the POLAR, the 1 K pot is operated in the so-called “single-shot mode”. This means that the 1 K pot is filled only once and then pumped until it is empty. This mode has several advantages: i) no adjustment of helium flow is necessary; ii) better temperature stability and thus, iii) better drift performance at low temperatures. The volume of the 1 K pot was designed to achieve an excellent long holding time of typ. 100 hours at T < 1.7 K. The 1 K pot can be combined with other options such as the 5 Tesla magnet, QPlus®, or the temperature variation package.

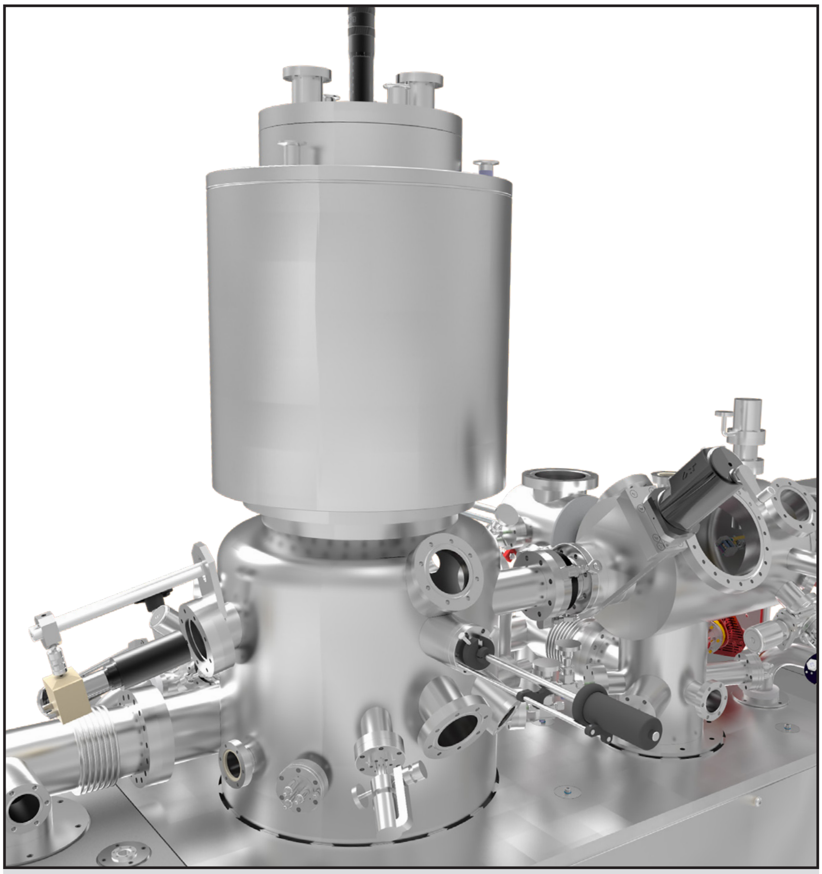
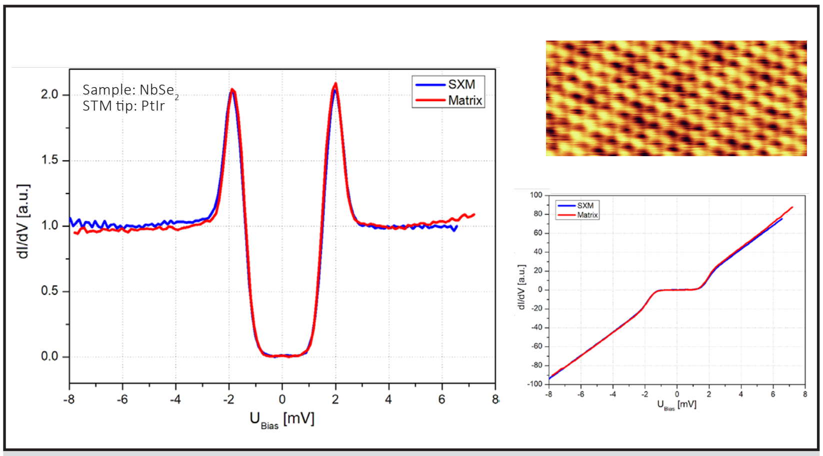


Figure: POLAR SPM lab with corresponding SPM chamber and sample preparation facility.



STM/STS at T = 1.49 K with POLAR 1 K pot: $NbSe_2$, A_{mod} = 100 μV_{rms}
Right image: STM topography on $NbSe_2$
Right spectrum: corresponding I-V spectra
The existence and the shape of the superconducting energy gap of $NbSe_2$ is clear proof of the achieved temperature of T < 1.7 K.

QPLUS® is a registered trademark of Prof. Franz J. Giessibl, Regensburg, Germany

ENHANCED ARPES EXPERIMENTS WITH PEAK AT SSRL

Seamless system integration using PEAK API

The ARPES beamline 5-2 (BL 5-2) in Stanford Synchrotron Radiation Lightsource (SSRL) in the US has been operating for four years after upgrading to a fully integrated control system using PEAK application programming interface (API). The PEAK API is specifically designed for seamless integration of the analyser with external hardware like beamline and manipulator. The integrated control system makes it easier to conduct various experiments that involve the control and synchronization of external hardware for enhanced experiments.

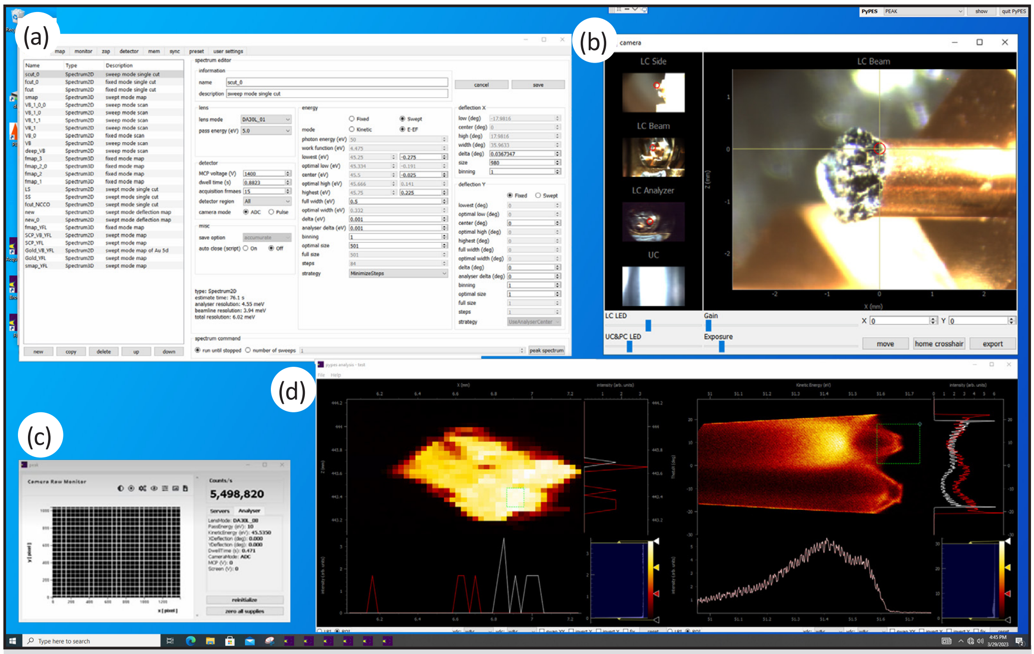


Figure: Graphical user interface of the integrated control system at BL 5-2 at SSRL. PEAK API enables seamless integration with external hardware. (a) Configuration of measurement scans and sequences. (b) Live camera image of a sample during measurements. (c) Live photoelectron spectra. Dummy image is shown in the figure. (d) 2D real-space µARPES intensity map (left), and band dispersion (right).

At BL 5-2, all the instruments related to photoelectron spectroscopy measurements can be fully controlled and synchronized from the integrated control system through graphical interface. External parameters are highly configurable to set up measurement scans and sequences, for example: 2D real-space mapping, temperature or excitation energy scan. Researchers can operate fully remote experiments without visiting the synchrotron, with the onsite help of the beamline staffs to transfer samples. Measured spectra are saved in an in-house HDF5 format, which includes metadata from the analyser and other hardware to facilitate data analysis.

The integrated control system at BL 5-2 is based on python, interfacing with Scienta Omicron analyser control (PEAK) and the beamline motion control (SPEC). The integrated control system controls hardware including the analyser, the low-temperature manipulator, the sample temperature controller, the beamline, and several cameras for the manipulator and the sample.

Beamline scientist Dr. Makoto Hashimoto:

– The users of our beamline experience the measurements at BL 5-2 to be efficient, thanks to the 2D real-space mapping and automated sample position optimization routines. It takes less than 30 minutes to find a good position on a sample and align the sample orientation before data acquisition.

– PEAK API enables such combination of analyser and manipulator control. PEAK API is very flexible, reliable, and stable. The python example code and the course provided by PEAK software development kit (SDK) are comprehensive and crucial for the efficient development of the software.

BL 5-2 at SSRL is designed for high-resolution ARPES in the energy range of 20-200 eV with full polarization control. The best spot size of the excitation source is 0.035 (H) X 0.007 (V) mm^2. The dedicated experimental end station is equipped with a DA30-L deflection analyser. This setup can perform ARPES experiments with a total energy resolution of a few meV and an angular resolution of ~0.1° while keeping the experimental geometry fixed to avoid matrix element effects.

PEAK SDK & Training Course supports analyser integration using PEAK API by providing software training course by the developers of PEAK, python example code, and PEAK demo license for development without analyser.

PEAK SLIT CONTROL UPGRADE

Optimal settings with remote control

In photoelectron spectroscopy measurements, there is always a trade-off between signal intensity and resolution. Optimising this balance is the key to obtaining smooth and sharp spectra within the shortest time possible. For hemispherical analysers, this trade-off is controlled by the selected entrance slit and pass energy.

PEAK Slit Control replaces manual slit changes at the analyser with a motorised and software-controlled slit. With the control of all analyser settings, easy and quick optimisation of signal intensity versus resolution is possible.

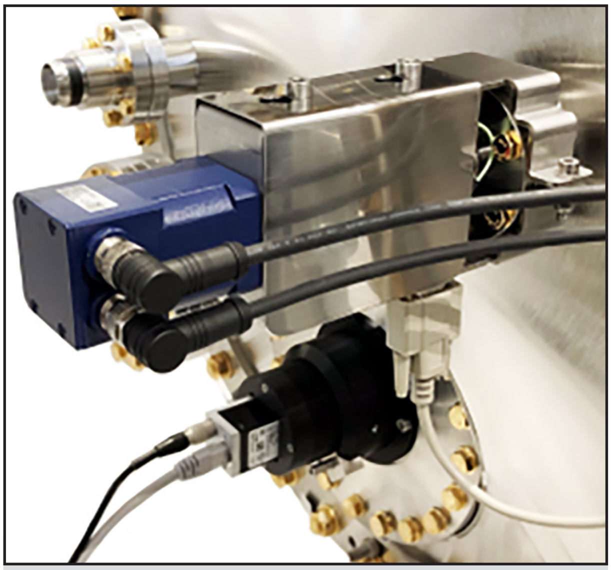
Remote control advantage

In hard x-ray photoelectron spectroscopy (HAXPES), the analyser is often in an experimental hutch. It is not easy to access during measurements, which makes it difficult to change the slit manually.

With PEAK Slit Control, the slit is controlled remotely from outside of the hutch, allowing users to adjust measurement settings at any given time.

Extended automatic sequences

Most experiments require a quick overview of the band structure, or of the elements present in the sample at high intensity. Later, measurements of small signatures and spectral features are made at much higher energy resolution. To do this efficiently, the slit must be changed other-wise the overview measurements would take too long or there would be insufficient energy resolution when studying small spectral features.



Motor and safety cover mounted to the slit selector feedthrough.

For similar sample types, optimal slit settings can be set-up within an extended automated sequence of overview and detailed measurements. With PEAK Slit control, the required slit changes are managed within the sequence and without manual intervention by users.

Reliable metadata

To repeat measurement conditions, or to estimate the analyser contribution to the energy resolution of a spectrum, it is crucial to have reliable information about the selected analyser slit.

PEAK Slit Control automatically detects the current slit using a position encoder. This information is then automatically stored in the metadata of each acquired spectrum. All analyser information is recorded in the metadata, so users do not need to rely on manual annotation of slit settings, which facilitates easier sharing and repeating of experimental conditions.

Upgrade

The PEAK Slit Control upgrade includes a motor assembly with stepper motor, a motor control box, the dedicated software license for the PEAK, Scienta Omicron´s electron spectroscopy control and acquisition software.

One of our service engineers installing the PEAK Slit Control to an ARPES Lab. Quick installation with no need for breaking vaccum.

All EW4000, HiPP-2/3, DA30-L, and DFS30 analysers can be upgraded with PEAK Slit Control.

The analyser must have PEAK for the PEAK Slit Control Upgrade.

Installation

The installation includes:

- Mounting of the motor control box on the frame of the spectrometer
- Mounting of the motor assembly onto the slit selector port of the spectrometer
- Cabling
- installation of software
- Execution of the slit position homing procedure

The installation is quick and can be done without breaking the vacuum.

Service visit is not required for the installation, but can be quoted from your local service representative.

HiPP LAB

Advanced APXPS measurements

The HiPPLab is a high-performance, laboratory-based system that includes a powerful small-spot monochromated X-ray source and a state-of-the-art HiPP-3 analyser. It is designed with ease of use, high count rate and stability as the number one priority, and will serve as an efficient, turnkey workhorse in both labs and shared facilities. The HiPPLab effectively tackles a wide range of scientific questions with applications covering catalysis, fuel cell analysis, battery research and involving the investigation of solid-gas phase, solid-liquid and gas-liquid interfaces.

The smart cone design of the HiPP-3 Analyser results in a stable and efficient performance of the system with high electron transmission that is suitable for multiple applications. Figure 2 shows measurement results from N_2 gas over Ag sample. The snap-shot mode allows for fast data acquisition during changes of system parameters. Figure 3 shows spectra raw-data from the drying process of liquid H_2O on Si.

The included MISTRAL software allows for system remote control of valves, temperature and turbo pumps as well as pressure reading (compare Figure 4). Together with the PEAK spectroscopy control and acquisition software the system becomes user intuitive and highly flexible to customer-wished adaptations. Possible system expansion include glove box, laser heating, electrochemical cells, gas reaction cell and prep. chamber.

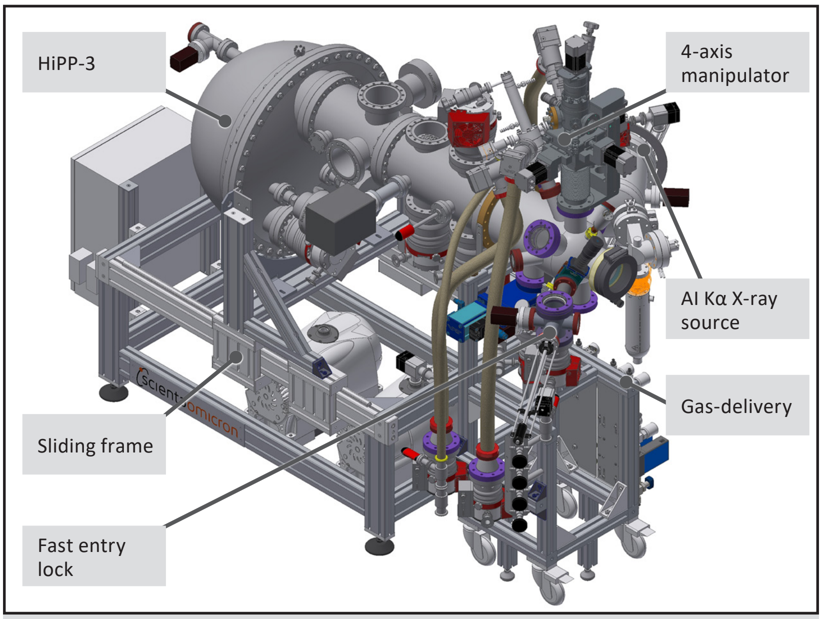


Figure 1: Example of a bespoke customer example where the system is equipped with a fast entry lock and a gas delivery system.

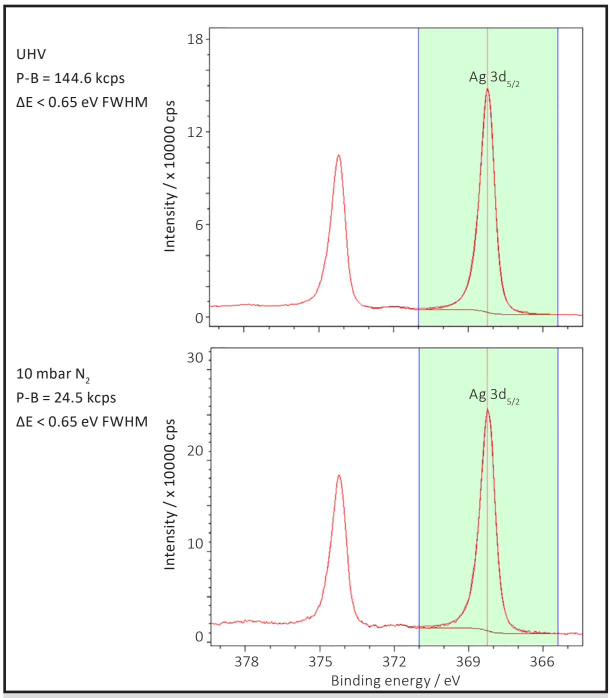


Figure 2: High quality Ag 3d APXPS spectra obtained using the HiPPLab. The spectra were acquired using 0.3 mm inlet aperture and N_2 background pressure. The figure indicates the peak (P) minus background (B) intensity being 144.6 kcps at UHV, 24.5 kcps at 10 mbar, 2.2 kcps at 25 mbar and 0.4 kcps at 35 mbar.

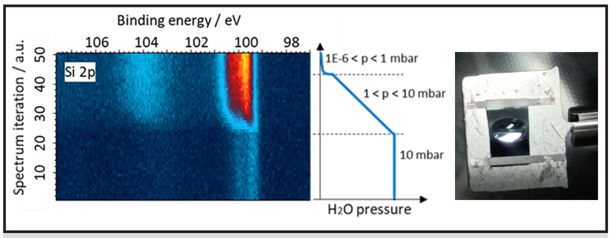


Figure 3: APXPS spectra following the drying process of a liquid water droplet over Si in H_2O background. Spectra are acquired using snap-shot mode with Swift acceleration and a 16 eV wide energy window and 60 sec. dwell time.

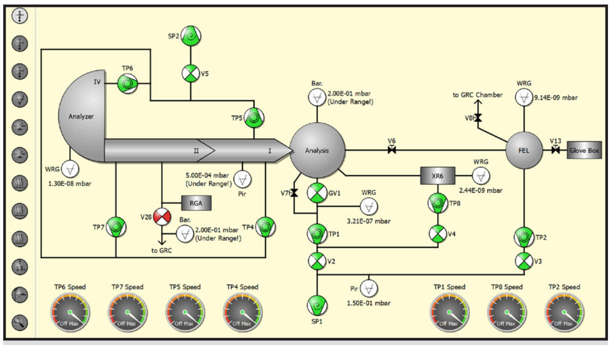


Figure 4: Example of a MISTRAL user interface to control and observe system parameters, e.g. gas feed, temperature, pump status.

ONGOING PARTNERSHIP BETWEEN SCIENTA OMICRON GMBH AND FOCUS GMBH

Highlights from the new NanoESCA MARIS system

In light of FOCUS GmbH's recent announcement of joining the Lab14 group, we want to assure our mutual customers that the collaborative relationship between Scienta Omicron GmbH and FOCUS GmbH remains unchanged. We are committed to continuing our close partnership, and no changes are anticipated to our relationship as a result of FOCUS GmbH's restructuring in ownership.

Scienta Omicron GmbH and **FOCUS GmbH** have a longstanding history of close collaboration, delivering cutting-edge instruments for research in surface science and nanotechnology. A notable outcome of the tight partnership between the two companies is the development of the NanoESCA product, a state-of-the-art PEEM (Photoemission Electron Microscopy) instrument, combining high lateral and momentum resolution with excellent spectroscopy performance.

NanoESCA MARIS

With its ability to perform both **M**omentum **a**nd **R**eal Space **I**maging **S**pectroscopy, the latest version of the instrument is called NanoESCA **MARIS**. Here, the lens system is completely modified in order to achieve a much improved angular / momentum resolution of 0.005 $Å^{-1}$ (as compared to its predecessor reaching a resolution of 0.015 $Å^{-1}$), while keeping the same excellent lateral resolution in real space of < 35 nm. The performance in momentum space is presented in Figure 1, displaying the band structure of a Au (111) single crystal measured at the Fermi level (Figures 1a and 1b). The Rashba split surface state is nicely resolved and fitted by two Lorentzian peaks (Figure 1c). The result shows that the inherent broadening of the spectral bands is considerably larger than the Gaussian broadening caused by the instrumental resolution.

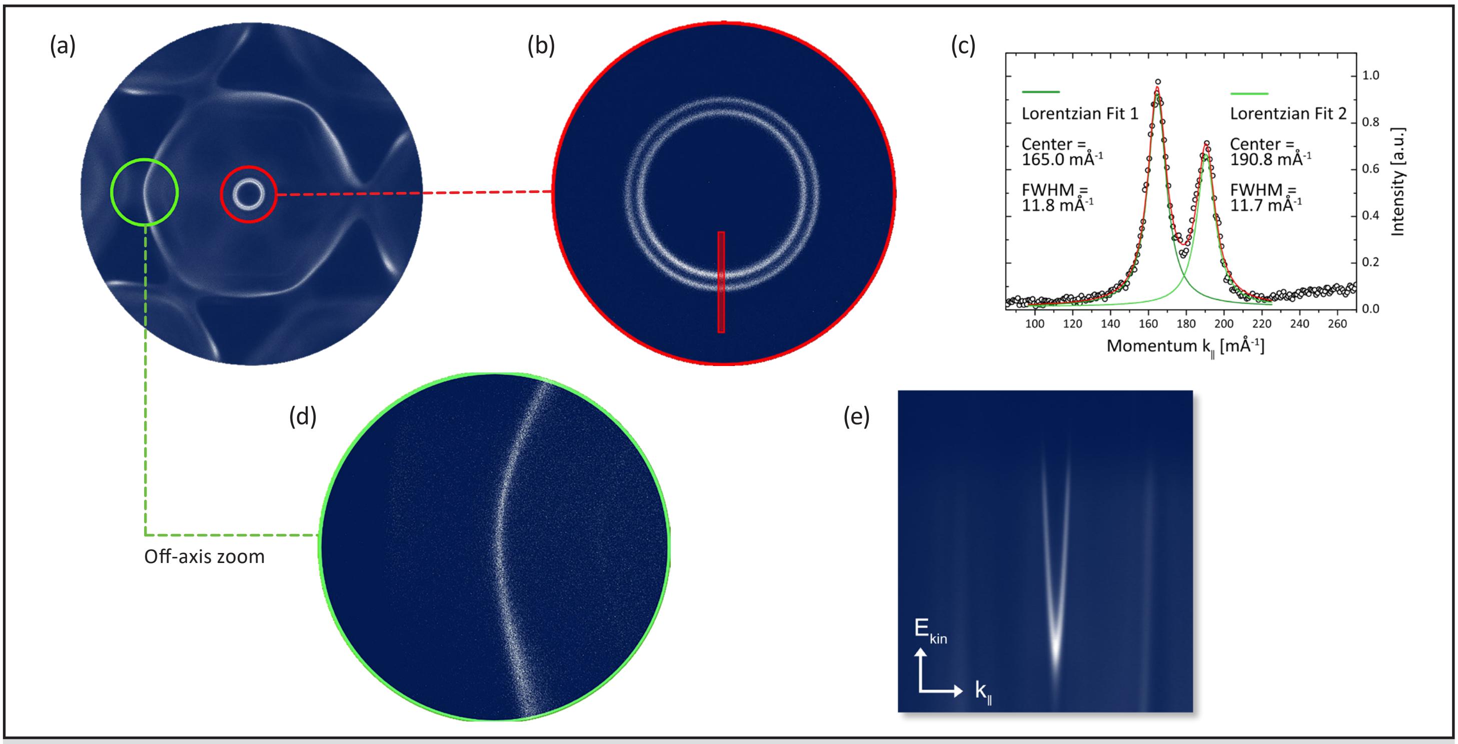


Figure 1: (a) Momentum Space Image of a Au (111) single crystal taken at the Fermi level, showing (a) more than one Brillouin zone and (b) a zoom onto the Rashba split surface state. (c) Line profile taken along the red line in (b) fitted with two Lorentzian peak functions. (d) Off-axis zoom onto the momentum space area enclosed by the green circle in panel (a). (e) Surface state of Au (111) measured with the Energy Dispersion Mode. All data are collected at a sample temperature of 45 K.

More often than not, spectral features away from the center of the Brillouin zone are of interest in ARPES experiments. As such, the new NanoESCA MARIS system allows to zoom onto any point in momentum space and collect band structural data around a desired k-point with high-resolution. The off-axis zooming capability is presented in Figure 1d.

In particular instances, there may be the need for a direct measurement of the Energy Dispersion Image. A new mode in the lens system – called the Energy Dispersion Mode – enables the projection of an energy vs. momentum cut directly onto the detector as shown in Figure 1e.

Low Temperature NanoESCA manipulator with Hexapod sample stage

For ultimate experimental resolution, it is always desired to cool down the sample. With the newly designed open cycle LHe cooled NanoESCA manipulator, we guarantee a sample temperature of < 10 K. The manipulator also hosts a 6-axis hexapod sample stage, enabling a proper in-situ alignment of the sample with respect to the optical axis of the microscope. The specifications of the low temperature hexapod sample stage are given in Figure 2.

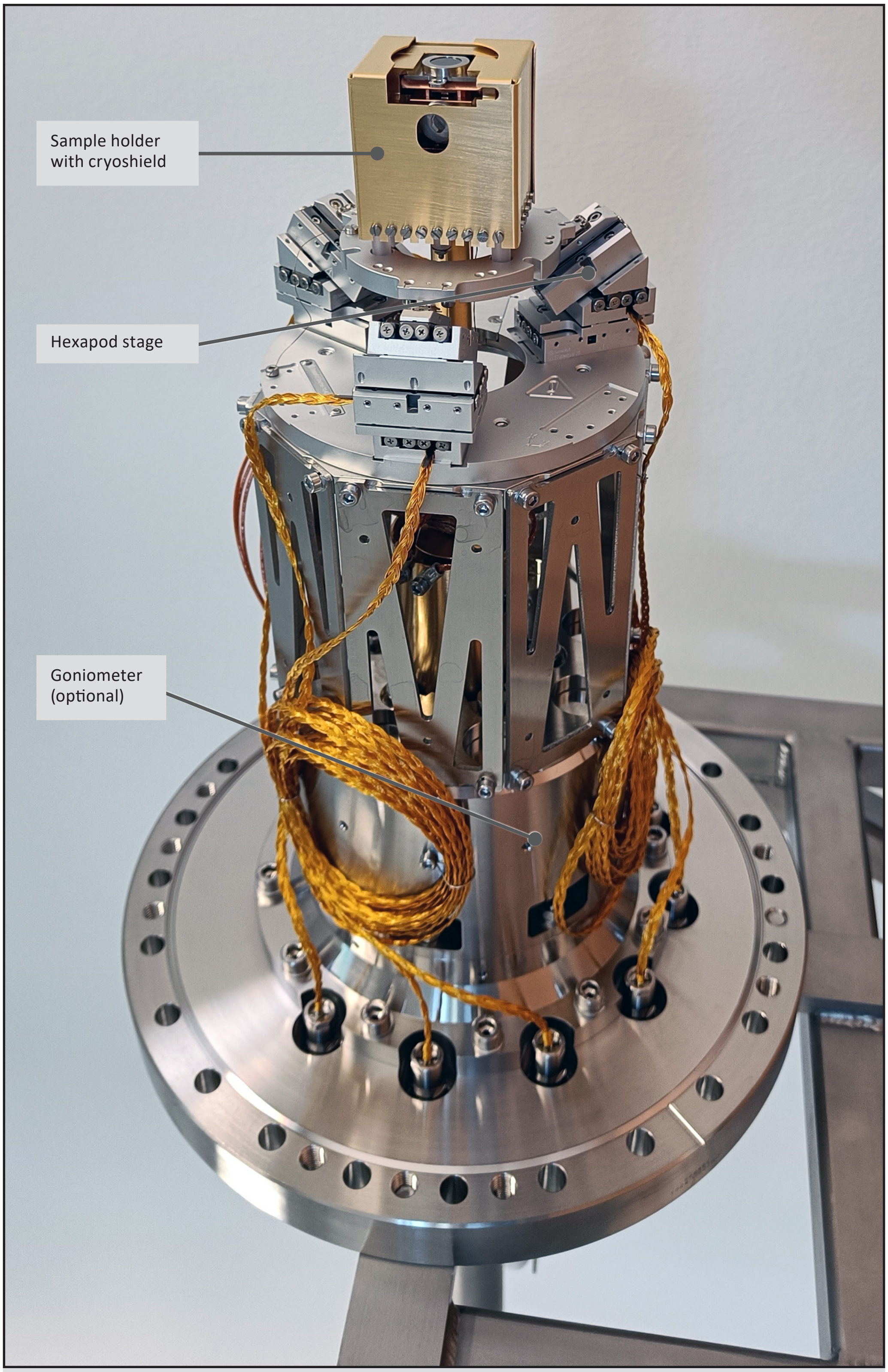
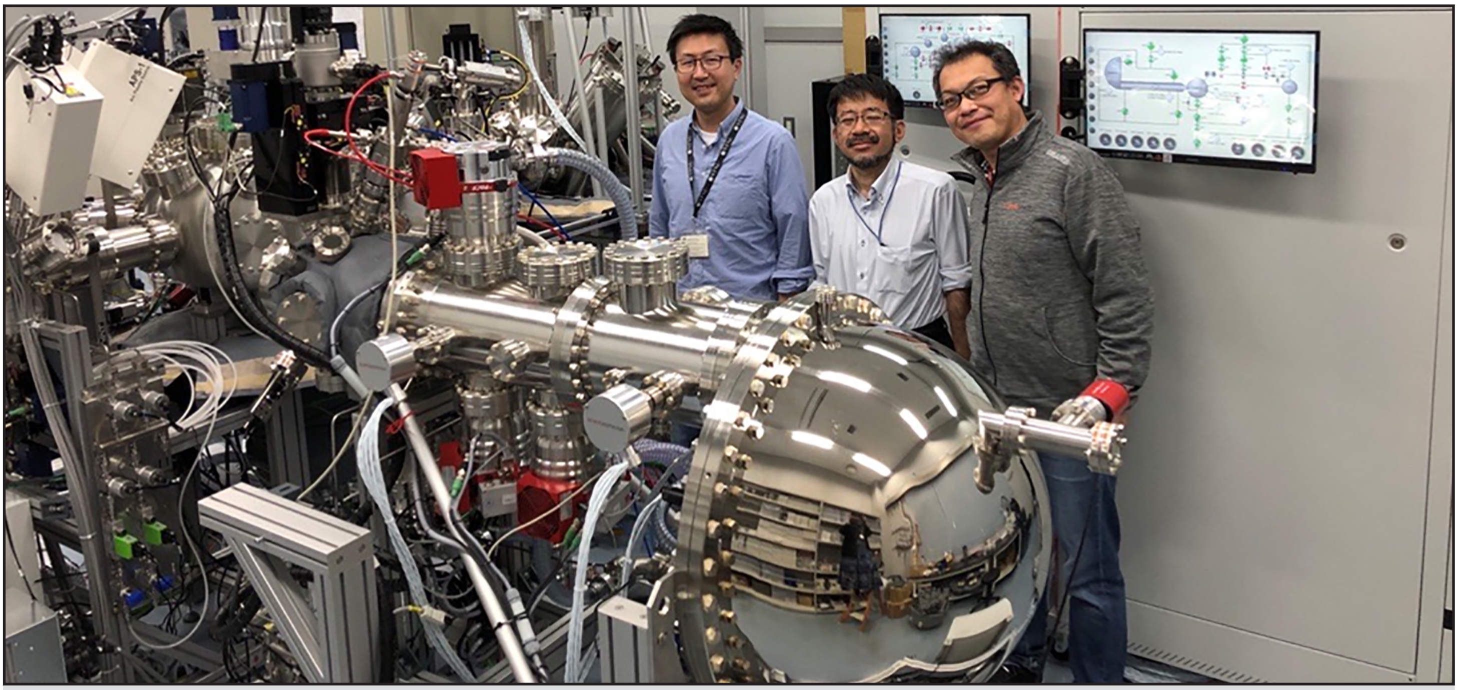


Figure 2: Low Temperature NanoESCA manipulator with hexapod sample stage.

- Temperature range < 10 K to 400 K
- x and y motion ±5 mm
- z motion > 5 mm
- tilt along x and y directions > ± 5°
- azimuthal rotation ± 10° (upgradeable to ± 60° with the addition of a goniometer)
- 4 electrical contacts to the sample (optional)

MATERIALS INNOVATION PLATFORM

APPES/XPS MIP system

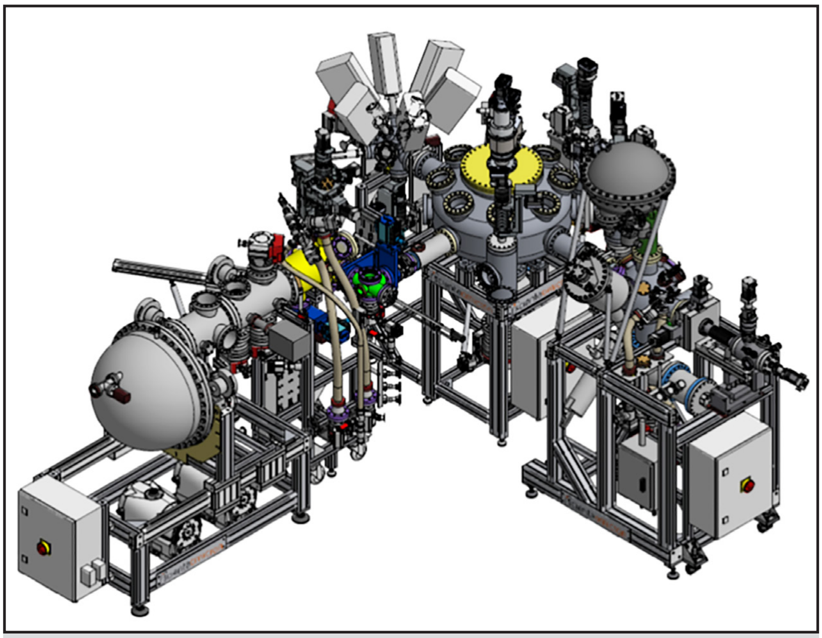


Installation photo: Dr. Tominaka (NIMS), Dr. Nakada (NIMS), Mr. Fukushima (SOKK) in front of MIP system at NIMS.

A APPES/XPS MIP system for catalyst research was successfully installed at the National Institute for Material Science (NIMS) in Japan at the end of March 2023.

Catalyst materials consist of multiple components and their performance is subject to a delicate interplay of element concentration, structure, porosity and the surrounding gasmixture. It is of utmost importance to improve the performance in a wide parameter space of temperature, gases and pressure. Dr. Tominaka and members of his group are developing a APPES/XPS system combined with AI/MI technology. Measured data of XPS/APPES on samples with different conditions will be stored in a Cloud-based data base. Dr. Tominaka's AI/MI technology in combination with Scienta Omicrons HiPPLab and XPS Lab will have major impact on the usage of AI for optimizing catalyst materials and involved sample growth processes.

The system consists of an analysis chamber equipped with DA20(R) XPS/UPS/ARPES analyser, a second analysis chamber equipped with HiPP-3 UPS/XPS, a sample growth chamber with arc plasma sources, a sample preparation chamber with a heater, and three load lock chambers. Each chamber is connected to the radial distribution chamber with a sample transfer robot. Samples are picked up from atmosphere to a load lock chamber and automatically selected and transferred to each chamber.



Schematic drawing of APPES/XPS MIP system.