FE-LEEM/PEEM P90 Series

COMPACT LOW ENERGY ELECTRON MICROSCOPE / PHOTOELECTRON EMISSION MICROSCOPE

SPECS

SPECS"

KEY FEATURES

- High Lateral Resolution
- Integrated Imaging Energy Filter
- Robust Sample Stage with Five Computer-Controllable Axes
- Sample Holder with Integrated Sample Heater
- Cold Field Emission Gun for LEEM



SPECS

SP*€*CS[™]

INNOVATION IN SURFACE SPECTROSCOPY AND MICROSCOPY SYSTEMS

SPECS leads the way in state-of-the-art technology for surface spectromicroscopy.

SPECS Surface Nano Analysis GmbH

SPECS has more than 150 employees at its headquarters in Berlin and its subsidiaries in Switzerland, USA and China. The company also has liaison offices in Spain and BeNeLux. Through the international sales channels customers in sixteen countries are supported. A team of scientists and engineers is involved in developing and producing scientific instruments for surface analysis, materials science and nanotechnology. Since the company has been founded in 1983





its success is based on a continuous gain in experience. SPECS experts are in close contact to a large number of customers and scientists around the world. SPECS is your essential partner in scientific instrumentation due to our focus on customer satisfaction, know-how and international contacts. Scientists all over the world can rely on SPECS high quality products and be inspired by the continuous development of new innovative solutions.

Packaging of a SPECS component after passing the final test

Final alignment of an UHV high precision sample manipulator

LEEM/PEEM

HIGH RESOLUTION ELECTRON MICROSCOPY OF SOLID STATE SURFACES

Just a stone's throw away from the first PEEM's birthplace, SPECS produces the state-of-the-art instruments with highest resolution.

History

Eighty years ago, **Ernst Brüche** developed the first **photoelectron emission microscope** (**PEEM**) in the AEG laboratories in Berlin. During the following decades the technique developed into a powerful tool for surface science allowing the study of surfaces with a resolution of a few nanometers under illumination of various light sources, such as UV lamps, lasers or synchrotron radiation. In combination with imaging energy filters laterally resolved photoelectron spectroscopic studies became feasible allowing the study of chemical composition at surfaces with highest spatial resolution.

Parallel to the improvements of electron optics Ernst Bauer developed the low energy electron microscopy (LEEM): By equipping the instrument with an electron source and a beam splitter, it became possible to illuminate the sample with a parallel electron beam normal to the surface. Those electrons are either elastically backscattered by the sample surface or inelastically scattered under going elementary excitations and producing secondary electrons. In both cases electrons are guided through the same electron optics and via the beam splitter towards the 2-dimensional detector. Depending on the primary energy of the incoming and scattered/emitted electrons, and if imaging is done in real or reciprocal space many different contrast mechanisms can be generated. While low energy electron diffraction (LEED) is an example for crystallographic studies, real space imaging can be done for instance in Mirror Electron Microscopy mode where the incoming electrons are reflected in front of the sample surface.

During the late 20th century, **Rudolf Tromp** at the **IBM Research Division** became aware of the possibilities of LEEM for the *in situ* observation of deposition processes or phase transitions and began to build a system by himself. The precision in instrument design and high manufacturing quality allowed this instrument to obtain the **highest lateral resolution** at the time.

As a result **SPECS Surface Nano Analysis GmbH**, situated in a historic AEG building just a few kilometers away from Brüche's former laboratory, decided to commercialize Tromp's instrument. By **continuous development** in collaboration with Rudolf Tromp at IBM, an **aberration correction** has been implemented, **pushing the resolution** limit even further to values **below two nanometers**.

Follow us exploring the nanoworld using the SPECS FE-LEEM P90.

FE-LEEM P90/PEEM P90

COMPLETE SYSTEM OPTIMIZED FOR HIGHEST-RESOLUTION MICROSCOPY WITH LOW ENERGIES

Fully integrated, stable system benefitting from almost vibration-free components and low maintenance

Introduction

FE-LEEM/PEEM P90 forms a state-of-the-art surface electron microscope reaching highest resolution in an easy-to-use compact design. Key features are fast specimen exchange, low vibration measurements, and *in situ* studies on dynamic surface processes. The base system is the PEEM P90 (without electron source) or the FE-LEEM P90 (equipped with a cold field emission electron source). Both are turnkey multichamber systems with an energy filter, sample storage and all necessary vacuum equipment.





The sophisticated energy filter enables imaging and local spectroscopy with an energy resolution down to 250 meV with a minimal impact on the high spatial resolution of the instrument.

The system can be upgraded with an optional aberration corrector for improved transmission and resolution. Additionally UV sources, such as a Deuterium lamp or a Helium lamp, for laterally resolved UPS studies, a preparation chamber and an SPECS Aarhus STM are possible options to add to the configuration. These upgrades are mounted on the same system frame for fast sample exchange and low-vibration operation of the whole system. Many different components for sample preparation can be installed in the analysis chamber for live observation of surface processes.

FE-LEEM P90

Electron Optics

The improved optical performance for highresolution imaging and spectroscopy is based on the energy filter and the reduction of electromagnetic influences (EMI) at the sample. The 90° magnetic deflector serves both as a beam splitter for the incoming and outgoing electron beams and as an imaging energy filter with an energy resolution of 1.7 eV and < 0.25 eV in imaging and spectroscopy mode, respectively.

All electron optical components are machined to highest precision and mounted in a stacking principle. This ensures the best possible mechanical alignment and allows for operation with a minimum number of electron optical deflection elements saving valuable time, because the operator does not have to perform additional system alignments. The self-shielded lens design provides an effective compensation of external stray fields.

As a result of the set-up a large field of view can be obtained ranging from sub- μ m to 100 μ m which is ideal for overview images as well as for more detailed surface analysis in e.g. domain-like structures.

Electron Source

The highest performance in illuminating a sample surface with electrons is achievable with a high-brightness cold field emission gun. The energy spread of < 300 meV is much smaller than in corresponding instruments equipped with thermionic or Schottky emitters. Furthermore the high transfer width leads to sharper images, diffraction spots and LEED patterns.

UV Sources

Alternative excitation sources can be offered giving flexibility to customized measurement techniques at different energy ranges. Light sources such as lasers, helium or deuterium lamps or synchrotron beams can be connected to multiple ports pointing at the sample.

For threshold photoemission microscopy studies the instrument is equipped with a Mercury UV source. Alternatively the Deuterium source DUVL 160 can be used.

The high performance Ultraviolet Source UVS 300 is ideally suited for ultraviolet photoelectron spectromicroscopy with the integrated energy filter. The UV source enables high resolution photoemission measurements with a small spot size and high flux density generated by a high density plasma (duoplasmatron principle). Using a special capillary this source can be focused down to a theoretical spot size of 500 micrometers. Differential pumping (50 l/s) enables operation at very low pressures in the analysis chamber $(\leq 1 \times 10^{-8} \text{ mbar})$. The UVS 300 can be mounted on a granite block for effective vibration isolation. For application examples please see Tromp *et al.*, J. Phys.: Condens. Matter 21 (2009), 314007.



UV source UVS 300

Sample Handling

Rapid sample handling by precise and reproducible positioning is key for good and reliable results, because the proper control of the sample position is mandatory for excellent overall microscope performance.

The sample stage in the FE-LEEM P90 is piezocontrolled and features five degrees of freedom:

- Distance sample objective lens
- 2 axes for lateral positioning
- 2 perpendicular tilt directions

All motors are equipped with position encoders and an external control pad or alternatively by the computer-controlled LEEM software. The sample stage is mounted directly on the objective lens for minimum sample drift and allows precise and reproducible positioning on a nanometer length scale.



Sample holders with integrated electron bombardment heater for sample caps (upper left) and for SH2/12 (upper right). The lower images show the respective sample stages The sample stage is available in two versions related to either the LEEM sample cap or the SPECS SH2/12. Each sample carrier is mounted to the sample stage via a corresponding sample holder which features a filament for electron bombardment sample heating up to 1500 K. For temperature or transport measurements or other applications four user-configurable electrical contacts are provided. The capabilities of the instrument can be extended to low temperatures by means of liquid nitrogen sample cooling, too. Both sample carriers feature simple and secure handling for reliable sample transfer.

The sample cap is dedicated to efficient local heating of the specimen and its sample stage is also available with additional azimuthal sample rotation. The SH2/12 sample holder is preconfigured with a thermocouple for temperature measurement and control during sample heating. It provides best compatibility with various SPECS equipment facilitating the combination of LEEM with complementary surface analysis techniques in multi-chamber systems.

Analysis Vacuum Chamber

Variations to change the sample environment from ultrahigh vacuum (UHV) to *in situ* conditions are possible at any time according to the goal of the research studies. The system consists of four independently pumped UHV chambers related to the analysis, electron optics, transfer and load lock. This way a vacuum of < 2 x 10⁻¹⁰ mbar can be achieved very fast in the analysis chamber. It is also possible to set the vacuum to elevated pressures to investigate decomposition processes on surfaces. The load lock/transfer chambers contain a storage possibility for several sample holders which are located *in vacuo*. The actual shape is tailored to the sample holder system used.



Load lock with sample storage

Software

To be able to tailor your experiments with full flexibility an intuitive and easy to learn software package is provided which enables every operator from novice to expert to drive the system to optimized performance. The latest software release of SPECS L5 is used for the simple control of the FE-LEEM P90 system including the electron gun, stage, camera and other accessories. To acquire proper data it allows the user both to individually set most microscope parameters independently (with very sensitive, dynamic ranges) as well as to script customized automation routines.

System Frame

For low vibration operation all components are installed on a single high stiffness stainless steel frame for efficient vibration isolation from the environment. For high-resolution measurements all mechanical pumps can be turned off. The aberration-corrected instrument FE-LEEM P90 AC additionally includes a reliable active vibration damping system. For installations in a noisy enviroment this option can also be added for the standard instruments.

Electronics

High stability and precise electronics improve the microscope performance tremendously, because high electron resolution studies demand very stable voltage and current supplies for the electron optics. SPECS has developed a set of high-stability power supplies needed for driving the resolution to the physical limits. All currents and voltages are computer-controlled by the LEEM/PEEM software package pre-configured on the measurement PC system.



FE-LEEM P90 system with frame and electronics

LEEM/PEEM

APPLICATIONS OF LEEM AND PEEM

Studying the surface structure benefits from the various applications.

Operation Modes

A variety of excitation processes gives access to the structural, chemical, electronic, and magnetic properties of the specimen surface. Using specific operation modes and illumination settings (choice between electrons or photons with variable energy) result in images in which the origin of the contrast formation reveals the actual properties of the investigated sample surface. While Photons may excite photoelectrons from core levels and valence states, electrons may result in elastically and inelastically scattered electrons. In addition, both kinds of excitation may result in Auger electrons and secondary electrons. Depending on the scientific question and complexity of the



problem complementary measurements can be applied. By simply switching between different operation modes correlated information from the same surface area are gained. The magnetic prism transfers both the LEEM image and the LEED pattern stigmatically, allowing routine switching between real image and diffraction. Both image and LEED pattern are transferred without the negative effects of chromatic dispersion, offering superior image and diffraction capabilities. The specific operational mode of Mirror Electron Microscopy (MEM), causes almost no sample damage which makes the LEEM/PEEM superior in this respect compared to many other conventional analysis instrumentation.



Spectroscopy PEEM: UVS 300 mounted to anlysis chamber (left)

View into analysis chamber: Objective lens with sample stage (right)

LEEM: Reflectivity Contrast



Different areas on the surface might show a difference in electron reflectivity depending on the surface material and structure. The reflectivity coefficient depends on the incident electron energy.

LEEM: Phase Contrast



Interference of electrons from alternating monolayers of the sample generates a vertical contrast, e.g. to make steps visible on the surface.

Mirror Electron Microscopy (MEM)



The electron energy is reduced such that the electrons return in the retarding field before hitting the sample surface. The contrast mechanism is based on local changes in the retarding field on the sample surface.

LEED



Diffraction pattern is formed in the backfocal plane of the objective lens. Using one lens in the projective column it is possible to image this LEED pattern on the screen.

Microdiffraction



By restricting the electron beam to a small area down to 200 nm it is possible to investigate LEED patterns of small regions, for example single islands or terraces.

Threshold PEEM



Electrons are excited with an UV light source. The contrast is based on local work function differences on the sample.

LEEM: Dark-Field Imaging



Using a single LEED spot in the intermediate plane for imaging by introducing an aperture in the diffraction plane. All areas on the surface that contribute to the existence of this spot

appear bright in the image, all other areas appear dark.

Energy-filtered PEEM



By inserting an additional aperture into the exit plane of the deflector prism and switching back to imaging mode, only photoelectrons with a specific energy are

used for imaging. This mode allows carrying out element-specific studies by selecting electrons with certain specific spectrosopic features for imaging.

Electron Energy Loss Spectroscopy (EELS)



When the sample is illuminated by the electron beam in spectroscopic mode the energy loss spectrum of the reflected electrons after interacting with the sample is displayed

on the screen. By studying the electron loss spectra with lateral resolution the local electronic structure of the sample can be investigated.

XMCD-PEEM



By illuminating the sample with circularlypolarized synchrotron radiation with variable photon energy the magnetic domain structure can be imaged via the X-Ray magnetic

circular dichroism (XMCD). The picture (courtesy of F. Nickel/Forschungszentrum Jülich) shows the domain pattern of an 8 µm Permalloy square element.

Angle-Resolved Photoelectron Spectroscopy (ARPES)



When the sample is illuminated with higher energy, either UV light from a helium or deuterium UV lamp or with X-rays, a spectrum of photoelectrons with a broad energy

distribution is generated. The dispersion of the 90° deflector prism provides the ability to obtain information about the kinetic energy of the electrons. By imaging the diffraction plane and inserting an entrance slit for the prism, angle-resolved photoemission experiments can be carried out.

Lateral Resolution of FE-LEEM P90

The image shown on the right has been taken on a clean Si(100)-2x1 sample at a kinetic energy of 3.5 eV. This surface usually consists of two domains rotated by 90° with respect to each other. Therefore, the LEED pattern detected from this surface is a superposition of two LEED patterns with one for each domain.

Operated in dark field imaging mode only electrons from one higher order LEED spot are transmitted to create the respective real space images. Hence, only the parts of the surface area (e.g. domain) contributing to this LEED spot appear bright in the image.

The step edges at the domain boundaries are used to determine the lateral resolution. The line profile has been created by integrating the data in the direction perpendicular to the steps within the marked area shown in the image.

The line profile shows, that the ultimate lateral resolution is well below 5 nm, down to 4.2 nm.





Line profile along a monolayer step of Si(100)

Dark field LEEM image of Si(100)

Lateral Resolution of PEEM P90

The ultimate lateral resolution of the SPECS PEEM P90 has been performed by imaging electrons emitted from a Cu/Ru(0001) sample excited by a Mercury UV-lamp. The Ru(0001) single crystal has been cleaned in situ in front of the microscope objective lens involving repeated flashing cycles at 1700°C in a background pressure of oxygen. The image shows a submonolayer of copper, deposited in situ at about 500°C on the clean Ru(0001) surface. The copper decorates the Ruthenium step edges. The smooth copper film quality has been achieved by slight postdeposition annealing at 800°C. The copper monolayer looks brighter because its work function is lower as in comparison to the clean Ruthenium terraces. The data shows an ultimate resolution of 7.2 nm.





Schematic experimental set-up of a PEEM P90



Cu on Ru(0001), field of view 20 μm

FE-LEEM P90 AC

ABERRATION CORRECTION FOR HIGHEST RESOLUTION LEEM

Significant improvement of transmission and resolution

Four-element electron mirror



A significant improvement of transmission and ultimate resolution is achieved by the aberration corrector. The spatial resolution of an electron microscope is normally limited by diffraction, spherical, chromatic higher-order aberrations.

Besides the energy spread of the electron beam the acceptance angle of the microscope is the other influencing parameter. For typical acceptance angles the resolution is dominated by the lowest-order spherical and chromatic aberrations of the objective lens. These can be compensated by a multi-element electron mirror carefully machined and calibrated. By eliminating the spherical and chromatic aberrations, the minimum resolution is significantly improved and reachable with bigger aperture sizes compared to non-corrected instruments which otherwise require very small apertures to get close to the same result. The resolution obtained is better by a factor of two and the transmission of the microscope is increased by a factor of eight.

The aberration correction consists of a second 90° beam splitter with transfer optics, that can be included initially or upgraded later. When passing the second beam splitter, the electron beam is deflected back to the electron mirror, then reflected towards the beam splitter where it is again deflected by 90° towards the projector column.

For proper operation of the aberration corrector all components are aligned as precisely as technically possible. The optimization result mainly from the high precision and quality manufacturing of the four-element mirror.



Schematic electron optics of the FE-LEEM P90 AC

Improved Lateral Resolution of Aberration Corrected FE-LEEM P90 AC

Chromatic and spherical aberrations of the objective lens are the limiting factors for the lateral resolution of the FE-LEEM/PEEM P90. These limitations can be overcome by reflecting the electrons emitted from a sample with a multi-element electron mirror. This improves the lateral resolution and transmission by a factor of 2 and 8, respectively.

Measurements have been performed on thin graphene layers grown on SiC(0001). By flashannealing the SiC substrate the graphene layers were grown *in situ* within the LEEM analysis chamber. The image has been taken from such a graphene layer. Areas with different numbers of carbon layers show different gray levels. Step contrast is seen in areas with the same layer number. The figure to the right shows a step profile and the area where it has been taken. The profile has been averaged over strips (five pixels wide) as indicated in the image by the blue box.





LEEM image of graphene covered SiC(0001)

System Integration

LEEM/PEEM INTEGRATION WITH OTHER METHODS AND COMPONENTS

FE-LEEM/PEEM P90 (AC) is a compact system. However an integration of many different analysis and preparation components from the SPECS Portfolio into the LEEM system concept is possible.

Integration into the compact system concept of FE-LEEM P90 (AC)

To a certain extend integrations into the compact system concepts are possible. Besides the pure integration of standard components also proven standard solutions are available to be integrated. The example demonstrates the integration of an STM Aarhus 150 HT chamber and a preparation chamber into a compact FE-LEEM P90 with Mercury and UVS 300 UV source, a port for a second electron source and an aberration corrector.



Components for the STM Aarhus 150 HT chamber

At highest resolution and stability the SPECS Scanning Tunneling Microscope (STM) allows for observation of processes on surfaces at an atomic scale. The miniaturized design of the STM Aarhus 150 (with the smallest mechanical loop between tip and surface) is unique in the field of commercially available STMs. Fastest scan rates can be achieved by this particular scanner head design. The tip may be cleaned and sharpened inside the STM with no necessity for tip replacement. A very time saving way of maintaining the quality of the measurements and results. The high temperature version of the STM Aarhus 150 allows for imaging of metals and semiconductors at elevated temperatures up to 1000°C by radiative heating during STM operation. For this reason the FE-LEEM P90 and the SPM Aarhus 150 HT perfectly complement each other with respect to an unambiguous interpretation of results. The STM, integrated into the system, allows for fast sample transfer between STM and LEEM without breaking the vacuum.



Components for the preparation chamber

Surface modification with subsequent observation of surface processes require the seamless integration of preparation and deposition methods. Sources like the Ion Source IQE 12/38, Plasma Atom Source MPS-ECR or the Multi Pocket Electron Beam Evaporator EBE-4 can be installed into the preparation chamber or alternatively into the LEEM/PEEM analysis chamber for true *in situ* studies.







Ion source IQE 12/38

Multi pocket electron beam evaporator EBE-4 (right image)

STM Aarhus 150 (left image)

MPS-ECS Plasma Source

System Integration

LEEM/PEEM INTEGRATION INTO MULTIMETHOD SYSTEM CONCEPTS

The compact LEEM/PEEM system itself can also be integrated into larger multimethod system concepts without compromising the performance. Thus an instrument platform with a unique combination of complementary techniques can be designed, with all parts provided from one manufacturer – SPECS.

Integration into Multimethod System Concepts

During the last decades many research topics have been identified, for which integrated UHV systems consisting of several analysis and preparation methods are indispensable. The integration of just a few methods can be realized by direct coupling of the respective analysis and preparation chambers. The LEEM is connected via a special decoupling device.

Complex Surface Nano Analysis System integrated with a Linear Transfer System



Larger system clusters with multiple methods or systems which grow over time and systems with both multi-user operation and customized flexible chambers are usually connected via transfer chambers. Inside those a convenient vacuum transfer enables the specimen to be transported from chamber to chamber in manual or automated manner. Also dedicated sample storages allow for easy and clear organization of the sample handling. SPECS has developed a special Linear Transfer System (SPECS LTS) combining compactness, easy connection and independent handling of the single satellite systems. The LTS is a highly reliable and wear-free sample transportation tool with no limitations in size and handling. Please contact SPECS for further information and consultancy on your special system configuration.

Technical Data

Specifications

FE-LEEM/PEEM P90 Se	eries

Resolution with FE-LEEM P90	Guaranteed: 5 nm, Achieved: 4.2 nm
Resolution with FE-LEEM P90 AC	Guaranteed: 2 nm, Achieved: 1.6 nm
Field of view	800 nm – 100 µm
Magnification	400x – 50000x
Energy resolution	Spectroscopy: < 250 meV Imaging: < 1.7 eV
Kinetic energy in the microscope	Typically 15 keV, scaleable down to 2 keV
Start energy	Up to 1000 eV
Base pressure	Better than 2 x 10 ⁻¹⁰ mbar
LEEM spot size	< 40 µm down to 200 nm with micro-diffraction aperture
Energy width of illumination beam	< 300 meV
Reproducibility of sample position	Better than 500 nm
Maximum temperature for imaging	1500 K





Dimensions of FE-LEEM P90 AC

SPECS Surface Nano Analysis GmbH Voltastrasse 5 13355 Berlin / Germany www.specs.com

T +49 30 46 78 24-0

F +49 30 46 42 083

E info@specs.com

