ElProScan

The ultimate scanning probe microscope system – going beyond classical SECM



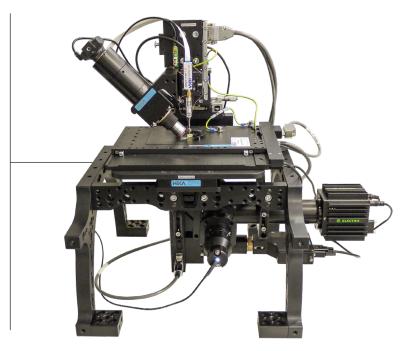




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The HEKA ElProScan is a unique electrochemical scanning probe microscope (e-SPM) system, which employs an ultramicroelectrode and/or nano-/micropipette as a scanning probe to perform *in situ* high-resolution microscopic imaging of local reactivity and topography of various types of samples and materials. ElProScans are offered in different models and various configurations to tackle a wide range of cutting edge and engineering applications.

Environmental Processes Nano-Electrochemistry Micro-Sensors Photoelectrochemical Processes Energy Conversion & Storage Corrosion Processes & Protective Coatings Micro-patterning & Micro-fabrication Neurochemical Imaging Electrophysiology & Single-Cell Imaging



Multifunctional Research Tool

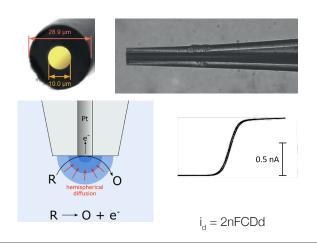
- Metals, alloys & cermet
- Active catalysts & carbon substrate
- Semiconductors
- Conductive polymers
- Hybrid nanostructures
- Biological cells & membranes
- Aqueous electrolyte & ionic liquid
- And more ...

ElProScan Supports State-of-the-Art e-SPM Techniques:

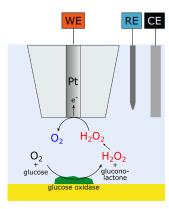
- Scanning Electrochemical Microscopy (SECM)
- Scanning Ion Conductance Microscopy (SICM)
- Scanning Electrochemical Cell Microscopy (SECCM)
- Scanning Microcapillary Contact Method (SMCM)
- Scanning Kelvin Probe (SKP) with µ-EIS
- Simultaneous Surface Topography Mapping
- Shear Force regulated Surface Tracking
- Synchronized Fluorescence Imaging
- Scanning Photoelectrochemical Microscopy (SPECM)
- Spatially-resolved Micro-spectroscopy (ECL/DFSS/SERS)

Scanning Electrochemical Microscopy (SECM), with decades of development and evolution, delivers a classical group of electroanalytical scanning probe techniques, capable of imaging and studying substrate topography and local reactivity with high spatial and electrochemical resolutions via the non-contact working electrode, an ultramicroelectrode with diameter ranging from approximately 20 nm to 25 µm. The ElProScan system has been considered by our worldwide users the best-in-class SECM, covering not only the classical working modes, but also many unique advanced scanning modes and combined techniques.

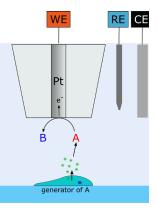
Ultramicroelectrodes



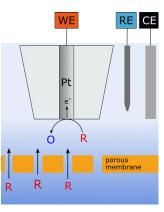
Classical SECM applications working with a microelectrode and diffusion-limited currents

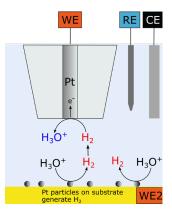


Electrochemical bio-sensors



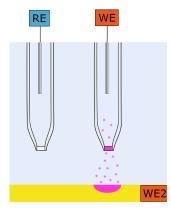
Imaging of live cells



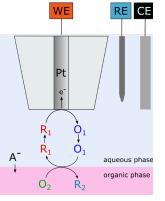


Molecular transport at porous membranes

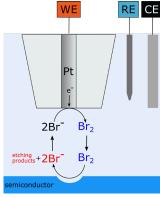




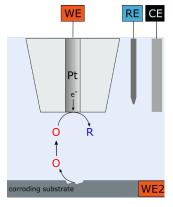
lon-selective potentiometric sensors



Liquid/liquid charge transfer

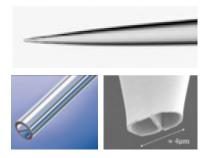


Micro-fabrication & patterning of surface



Corrosion mechanisms

In addition to SECM with a scanning microelectrode as probe, a unique group of e-SPM techniques employ micro-/nanopipettes as a scanning probe, which is regulated via detecting its ionic current at the tip end for distance-control between the probe and substrate to achieve nanoscale topographical mapping of soft and delicate surfaces. Such a micro-/nanopipette probe can also be modified and used as a bi-functional probe to detect faradaic current (i.e. surface reactivity) from the tip end, which is either submerged in bifunctional electrolyte or operated in a controlled gas environment.

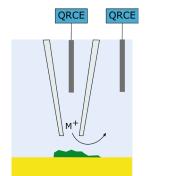


Examples of single-barrel and dual-barrel micropipettes

SICM/SECCM applications working with a micro-/nanopipette and ionic currents

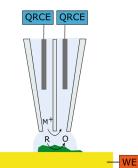
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Scanning Ion Conductance Microscopy (SICM)





Combined SECM/SICM

Scanning Microcapillary Contact Method (SMCM)

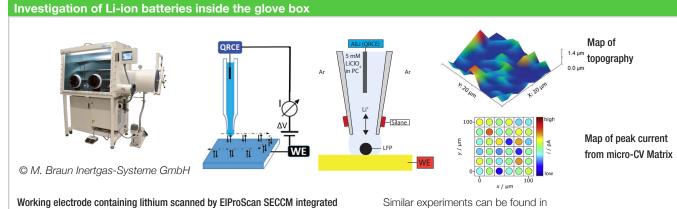
Scanning Electrochemical Cell Microscopy (SECCM)

Probe scanned within bulk electrolyte

- Non-destructive high-resolution topography imaging
- Combined SICM Fluorescence Microscopy
- Targeted SICM Patch Clamping
- Combined SICM-SECM for simultaneous imaging of surface topography and reactivity

Probe with a micro-/nanodroplet cell under controlled gas environment

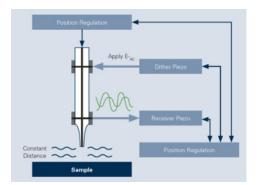
- Spatially resolved local electrochemical mapping:
- Micro-CV & micro-fabrication for conductive samples
- Combined imaging of electrocatalysis activities and nanoscale topography for nanomaterials

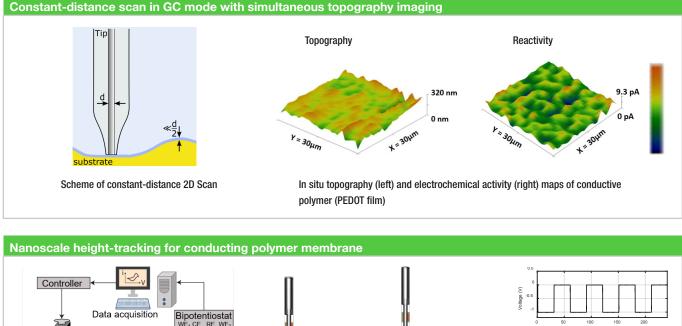


in a glove box.

Similar experiments can be found in Snowden et al. Journal of Power Sources, 325 (2016) 682-689 To overcome the drawbacks in traditional constant-height mode of SECM, Shear Force based height regulation has been proven to be the most effective and reliable mechanism for the advanced constant - distance scan mode, in which the probe scans along the sample's contour keeping a constant tip-to-substrate distance and the tip current signals are thus decoupled from the substrate's topographical complications.

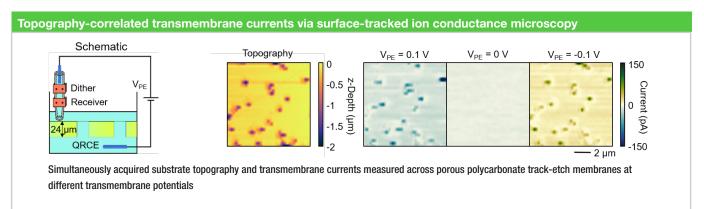
The HEKA Shear Force Extension allows simultaneous recording of topography and current signals in constant-distance regulation of SECM and SICM/SECCM.





SECM tip Pt Δz Ag/AgC و و 0 •• • Oxidiz d PPv(DBS)⊕ Substrate XY stage Electrolyte containing Measuring volumetric strains in conducting polymers between redox mediator (100 mM KCl + 1mM FcMeOH) oxidized and reduced states

Methods outlined in Northcutt, R. G., Heinemann, C., & Sundaresan, V. B. (2016) Physical Chemistry Chemical Physics, 18(26), 17366-17372.

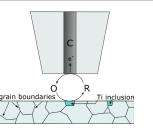


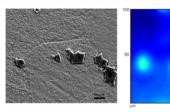
Methods outlined in Venkatesh, V., Heinemann, C., & Sundaresan, V. B. (2019). Micron, 120, 57-65.

In material sciences, the study of degradation and corrosion processes is of interest. SECM as well as micropipette techniques have been applied to investigate corrosion kinetics (generation-collection mode of SECM), local corrosion potentials (SECCM) and localized impedance measurements (SMCM). The combination with Shear Force sensing allows constant-distance scans.

Studying localized corrosion processes at alloys by SECM

The addition of Ti to ferritic stainless steel inhibits intergranular corrosion but takes part in the initiation of other localized corrosion processes. Spots of high reactivity in SECM images were attributed to Ti-rich inclusions. The localization of alloy elements will help understand localized corrosion resistance.



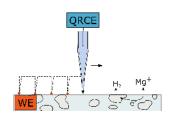


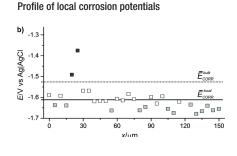
Scanning Electron Microscopy image (left) and SECM (right) image map of similar sites on the substrate. The local hotspots correspond to the conductive inclusions.

Gateman, S. M., Stephens, L. I., Perry, S. C., Lacasse, R., Schulz, R., & Mauzeroll, J. (2018) npj Materials Degradation, 2(1), 5.

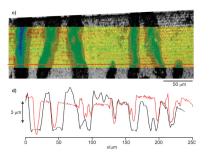
Mapping in situ corrosion potentials and surface profile by SECCM

Corroded Mg alloys were investigated for topography variations and local corrosion rate.

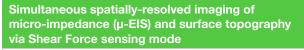


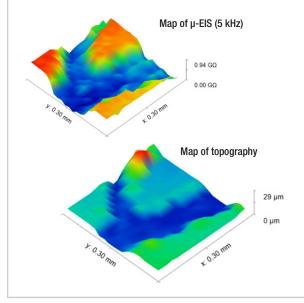


Height profile of corrosion surface



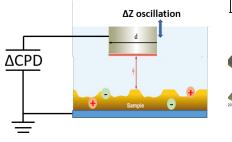
Dauphin-Ducharme et al. Faraday Discussion 2015, 180, 331-345 – Reproduced by permission of The Royal Society of Chemistry

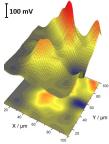




Scanning Kelvin Probe (SKP)

- A material's work function is an extremely sensitive indicator of surface conditions and is affected by adsorbed or evaporated films or species, surface reconstruction, surface charging, oxide layer imperfections, local and/or bulk corrosion processes.
- HEKA's SKP scans a vibrating capacitor probe to image work function at true micron scale.





Flexible constant-height or constant-distance mode Map of work function from a corroding surface

The ElProScan system consists of three main modules: the mechanical positioning stage, the dual-channel electrochemical workstation, and a real-time controller. Each of these functional modules is designed to be easily customized to meet your particular application requirements. Model-specific modules include a synchronized optical imaging system and the Shear Force extension.

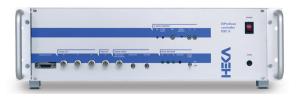
High-Precision Positioning System

- The EIProScan 3 platform utilizes the highest quality closed-loop controlled multi-axis motorized positioning system. A linear encoder (2.5 nm resolution) at each axis allows a real-time position control with closed loop regulation to eliminate backlashes.
- The XY-motor stage manipulates samples in the XY-direction, and the probe (either an ultramicroelectrode or a nano-/micro-capillary probe) is scanned vertically by a Z-piezo/motor assembly (with 1.5 nm resolution in height measurement).



ELP 3 XYZ positioning scan stage with inverted motorized focus axis for optical imaging.

The New Generation of ElProScan Controller (ESC 5)



ESC 5 Controller for ElProScan

- The new ESC 5 Controller may be equipped with hardware lock-in electronics to empower the latest HEKA Shear Force Extension (SFU 3).
- Controller employs on-board computer system to process and integrate real-time positioning data with electrochemical signals online.
- The design allows the ESC 5 Controller to interface with and gain control
 of a wide range of external analog-driven piezoelectric stages, thus
 offering a cost-effective upgrade solution to users' existing non-HEKA
 scan stage.

Cutting-Edge Electrochemical Resolution for Nanoelectrochemistry

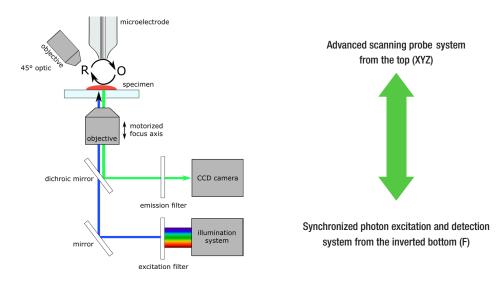


- Low noise: 3.5 fA @ 15 Hz and 31 fA @ 1 kHz
- High resolution: 0.15 fA in 5pA range
- · High bandwidth and fast sampling rate

PG 618 USB USB Ultra Bipotentiostat/Galvanostat

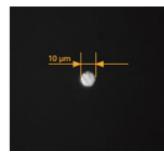
- The world's 1st asymmetric dual-channel bipotentiostat with different current ranges for each channel, specially designed to support noise-free ultralow potential and current sensing experiments
- Built-in 16-bit 200 kHz AD/DA interface with 5 µs shortest sample interval
- · User-configurable hardware filters tailored for fine tuning of electric signals
- Additional auxiliary I/O ports empower the control of external equipment

HEKA's ElProScan allows the integration of an inverse microscope into the scanning platform enabling a wide range of extended techniques.



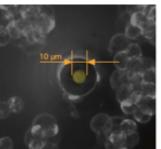
An illustration of ELP 3 system with epi-fluorescence optics

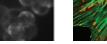
The Unique Modular Microscope Provides an Extendable Platform



Reflection Image

Transmission Image



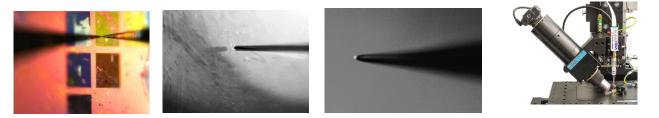


Combined Image

Fluorescence Image

- The system's built-in inverted modular microscope supports bright/dark field imaging, as well as many types of high-end fluorescence light source (synchronized LED/Xeon/Laser).
- The uniquely configured optical train features modular designs to synchronize tip-scanning with various optical microscopy and micro-spectroscopy techniques.
- The inverted optical axis remains in focus with the collective tip/sample-scanning and hence the optical measurements are synchronized with SPM, which ultimately provides multi-dimensional 3D/4D/5D data and microscopy images within one scan experiment.

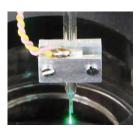
The Convenient Oblique 45° Imaging System



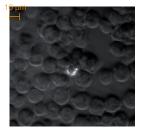
• The high-speed HD camera may record videos and triggered photos of tip and substrate during scanning probe experiments, which facilitates prepositioning and documenting relative positions of tip and sample.

- SmartLUX software allows the user to perform optical image acquisition and electrochemical recordings simultaneously. The timing of fluorescence excitation by the light source, the camera exposure and the electrochemical data acquisitions are all precisely synchronized by the software.
- SmartLUX is an ongoing HEKA research & development project. The current product features high temporal resolution and exact correlation among fluorescence data and electrophysiological & electrochemical data.

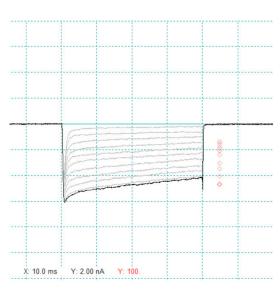
Correlation between fluorescence and electrochemical current signal



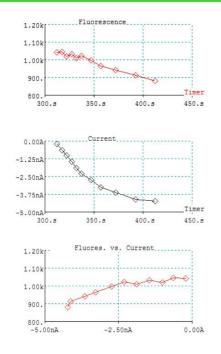
Epi-fluorescence illumination of the microelectrode tip with monochromatic light through the objective



A combination of a fluorescence image with transmitted light image of the mitochondria of one transfected T-cell.

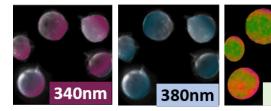


Software oscilloscope window simultaneously displays the timing of the optical measurements, image acquisition and the electrochemical (E/I) signals, all of which are synchronized with a temporal resolution on the millisecond scale.



Three Online Analysis plots show the exact correlation of measured fluorescence intensities with electrochemical current signals.

Investigation of calcium concentration



High Ca²⁺

Low Ca2+

EIProScan may be tailored to offer a special hardware package to tackle the most challenging requirements of ratiometric imaging.

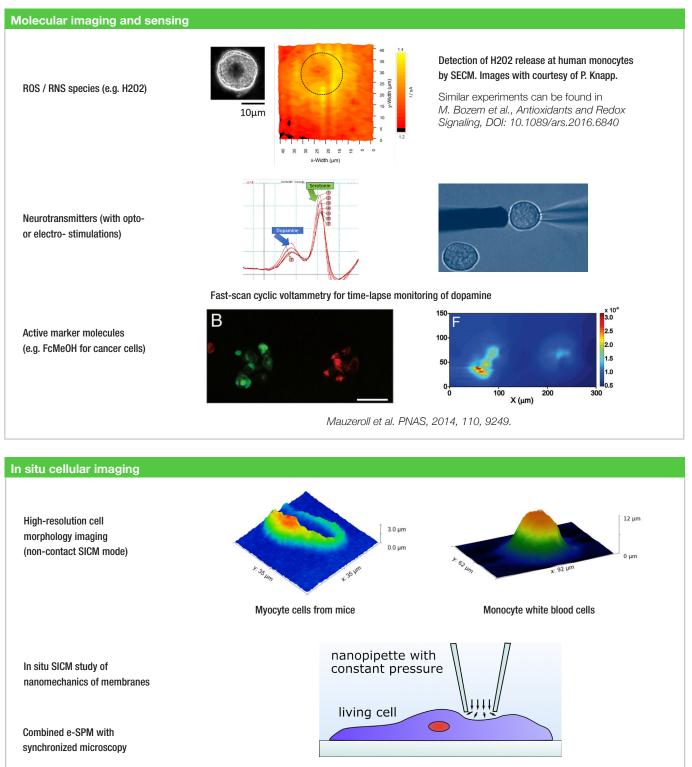
Snapshots (top-left) from a time lapse calcium imaging experiment using the ratiometric calcium indicator Fura-2.

All ratiometric methods have in common that the intensity of emitted light is measured twice and a ratio (R) of these intensities is calculated to represent relative concentrations of the specific ions.

Ratio

Single-Cell Electroanalytical Imaging Workstation

HEKA's ELP 3 system pioneered in many of the world's first imaging applications with its unique integration of synchronized optical microscopy imaging and high-resolution low-noise electrochemical imaging techniques. From molecular sensing to cellular imaging technologies, ELP 3 system has become the ultimate workstation platform defining the latest standard for a new generation of single-cell electroanalytical imaging workstations in interdisciplinary biophysical, bioanalytical, electrochemical and neurochemical applications.



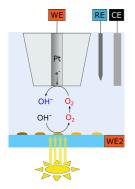
Similar experiments can be found in T. E. Schaffer, Anal. Chem. 2013, 85, 6988-6994.

Scanning Photoelectrochemical Microscopy (SPECM)

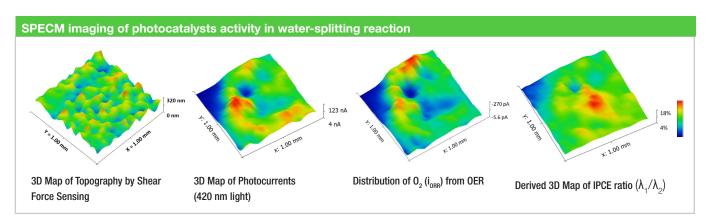
In 2016, HEKA launched the first commercial SPECM with combined SECM/SICM/SECCM and in situ synchronized photo excitation for microscopic study and imaging of photoelectrochemical processes on the micron and sub-micron scale.

The innovative SPECM works for a wide range of materials and applications:

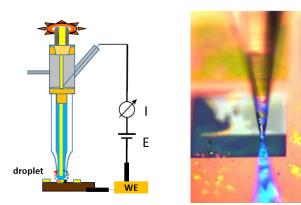
- Inorganic Semiconductors
- Semiconducting Polymers
- Hybrid Nanostructures
- Organic Photovoltaic Materials
- Solar to Electricity Conversion (solar cells)
- Solar to Chemical Energy Conversion (Water splitting and CO, reduction)
- Photosterilisation, Self-Cleaning Surfaces
- Environmental (air and water) Remediation

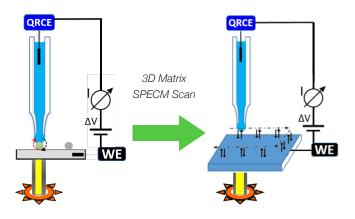


- Simultaneous mapping of microscopic distribution of photocurrent, IPCE/QE, photo-sensitive products or intermediates, and high-resolution surface topography within one scan.
- · Automatic multi-wavelength switching synchronized with dual-channel current recordings



• Innovative SPECM setup supports optical fiber couplings with SECCM microdroplet cell or SICM mode using HEKA's Opto-Pipette Holder.



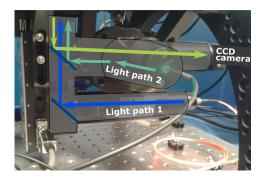


Top-illumination via Opto-Pipette in microdroplet cell mode (left) and top camera view of Opto-Micropipette tip (right) and mirror image from a wafer sample

Inverted illumination in 3D matrix scan in SECCM mode

The Innovative ELP 3 for Combined Microscopy and Micro-Spectroscopy

The 2019 ELP 3 in SPECM-FL optical configuration features a seamless integration of epi-fluorescence optics with optical fiber-coupled excitation/detection light paths, targeting a variety of optical microscopy and microscopy that can be integrated and synchronized with e-SPM techniques.



Secondary Light Source for Fluorescence Excitation

CCD Spectro-Photometer (or Fiber Excitation Source)



Light path 2

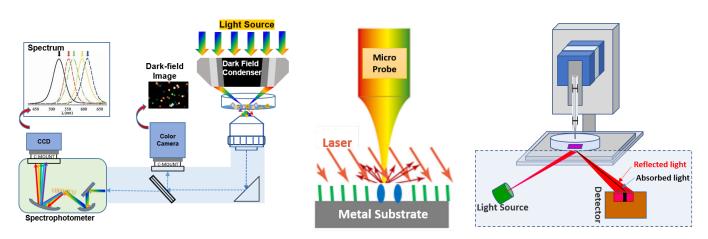


Light path 1

Illustrative photo shows ELP 3 inverted dual-light path under the XY-scan stage

The SPECM-FL design facilitates in situ microscopic studies of quantum dots or catalysts with synergistic localized energy and electron transfer under two individual wavelengths of light.

The inverted camera view with light shining on substrate (dia. 50 µm spot via light guide vs. 1 µm spot via optical fiber)

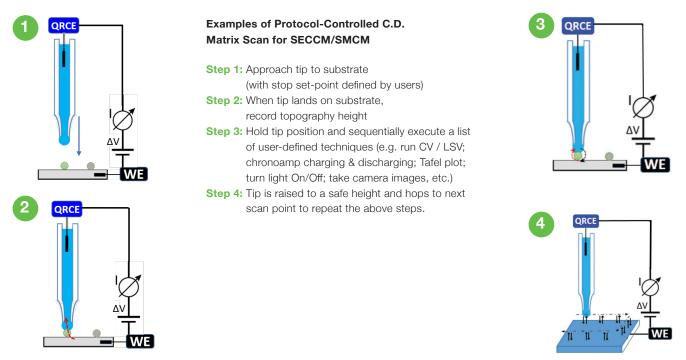


Multi-functional platform for Synchronized Micro-Spectroscopy Imaging

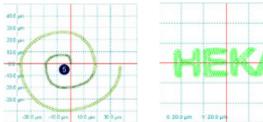
The ELP 3 combined platform allows nano-scale imaging and analysis of localized micro-spectroscopy signals from absorption/reflection, surface plasmon resonance (LSPR), DFSS or ECL from individual particles or molecular entities for development of biological nano-sensors.

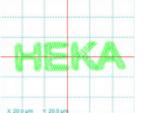
HEKA software for EIProScan (POTMASTER and PATCHMASTER) has been developed to provide powerful and flexible data acquisition, data analysis, and plotting functions. Always compatible with all versions of Windows (XP - Win10) and MAC OS, it supports the world's most comprehensive and up-to-date electrochemical scanning probe techniques, and additionally, the following unique features are available to empower advanced customization and automation of one's own experiments.

User-defined protocol scripts for automating a complex experiment



User-directed template scan for surface patterning and microfabrication





ElProScan: Scan	- • •
Scan Mode Template Scan Start scan	Stop Go back
(Load Template	Slope (X) 0.00
Protocol Select Prot.: CV-Method	Slope (Y) 0.00
Samples 0	1

Along each scan path, drawn by users in the imported template graph, the software applies a list of user-defined protocol scripts, such as user-defined "etching" or "electrodeposition".

- Users may draw any scan path/patterns and save as vector graphics, which may be imported in Potmaster and used as the probe scan template. The spatial size and resolution of the scan pattern are completely user-defined and can be flexibly matched to the probe size.
- At each of the predefined spatial points, the software can apply any combination of predefined electrochemical experiments and tipscanning motions (e.g. pulsed etching, electrodeposition, 3D probe movement, etc.).
- With one mouse click, the template scan fulfils the functions of surface patterning and/or micro fabrication, and even microscopic 3D printing.

The following specifications are only applicable to a standard ELP-3 SPECM configuration.

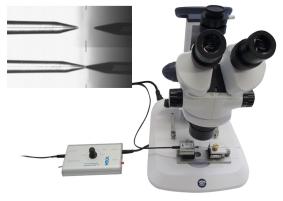
Components and Items		Technical Specifications
Positioning System with Integrated Microscope Optics	XY/Z/F 4D positioning system	4-Axis DC servo motors with Z-axis piezoelectric system (XY-axis carries sample in scanning; Z-axis carries microprobe, and F-axis drives objective lenses for precise focus).
	Resolution of X/Y/Z Axis	Linear Encoder of each motor axis = 2.5 nm resolution (all closed-loop controlled); 1.5 nm resolution for the Z-axis piezo module; XY-scan resolution = 10 nm with 4 times oversampling algorithm to ensure accuracy
	X/Y/Z/F Axis Travel Range	Automatic motor-scan range: X = 100 mm, Y = 75 mm, Z and F = 50 mm. Z-axis piezo range = 100 μ m (closed-loop controlled) A manual translator unit extends XY range to additional 12.5 mm.
	4D External Joystick	For X/Y/Z/F manual control with sub-micron accuracy; may operate in precise-slow motion and coarse-fast motion.
	Integrated Microscope Optics	The inverted optical train contains a motorized focus drive, epi-fluorescence optics in Kohler configuration (with a variable field stop) and special coupling to a liquid light guide, camera port with C-mount, an optional filter cube holder, and slide-in beam splitter and a mirror cube. Additional fixed-spot size illumination path with coupling to a FC/PC fiber may be added. Optional objectives (4x up to 100x) can be mounted in a 6-position turret nose piece. Microscope optics transmits light of wavelength above 330 nm.
	Optional Upgrade	Top 45° camera system may record and view the microprobe and sample surface in prepositioning, greatly facilitating Z/F-axes alignment and pre-scan preparations.
	Voltage Range / Resolution	\pm 10V (in single amplifier mode) / 610 nV (compliance $\pm 12V$)
	Current Ranges	± 20 nA to ± 100 mA (Amp-1); ± 5 pA to ± 2 μA (Amp-2; total 18 ranges available)
Bipotentiostat	Max. Current Resolution	0.15 fA in 5pA range (Amp-2); 0.61 pA in 20nA range (Amp-1)
WorkStation	Noise in Current	RMS value < 3.5 fA (at 15 Hz bandwidth in 5 pA range of Amp-2)
	DAC Interface	16-bit / 5 µs fastest pulse / 200 kHz sampling rate
	Optional Upgrade	External EIS measurement module (10 µHz – 2 MHz)
Photo- Excitation System (optional)	Synchronized Multi- Wavelength Excitation System	Standard package includes: 300W Xenon arc lamp, shuttered 10-position filter wheel, fused silica light guide, 10 bandpass filters with 10nm bandwidth in the range of 350nm to 800nm, 1 neutral density filter).
	Light Guide to Fiber Coupler Kit	Used for connecting a light guide with 5mm OD and 20mm length fitting to a SMA type optical fiber for the fixed spot-size illumination path.
	Opto-Pipette Holder Kit	A special pipette holder with a straight optical port (SMA type) used for coupling a HEKA cannula to an optical fiber for combined top-illumination in SICM/SECCM experiments.
	Optional Upgrade	Special customization is available for adding Synchronized Fluorescence Imaging module. Additionally, LED and Laser light sources are available.

Comp	onents and Items	Technical Specifications	
Shear Force Extension	Mechanism	AC modulation of piezo stimulation amplitude and phase	
	Topographical measurement	Height resolution = 1.5 nm; simultaneously obtained in an independent data trace with tip current recorded in Constant Distance mode.	
	Stimulation Amplitude and Frequency	0 V to 1 V (sine wave); selectable frequency = 100 kHz to 1 MHz	
	Constant Distance regulation	Maximum 5 μm (with 1.5 nm as smallest calibration step)	
POTMASTER Software	SECM Scan Mode	 Automatic and manual Probe Approach Curves (1nm/s up to 50 μm/s on Z-piezo) Constant-Height 2D/3D Scan at constant velocity (10 nm/s slowest, with slope compensation and user-defined scan axes) Constant-Distance 2D/3D Scan (available modes: Shear Force Hopping / Surface-Tracking / DC Hopping / Z-modulated Hopping for SECM/SICM/SECCM/SMCM) Template Scan (user-defined micro-etching / micro-deposition / micro-3D printing; XY scan cycles available in zigzag or alternative directions) 2D/3D Protocol-Controlled Matrix Scan (each scan point allows running any combination of experiment waveforms and sequence). 	
	SECM Working Mode	 Feedback Mode Generation-Collection Mode Redox-Competition Mode Surface Interrogation / Titration Mode Mediator-Free direct Mode Non-Hopping Continuous Surface Tracking Mode Depth Scan (X vs. Z scans) Mode AC-SECM Mode 	
	Supported Electrochemical Techniques	 Classical Voltammetry: Open Circuit Potential; Cyclic Voltammetry, FSCV (up to 2 kV/s), Linear Sweep Voltammetry; Staircase (Linear and Cyclic) Voltammetry, Chronoamperometry, Chronocoulometry, Chronopotentiometry, Normal Pulse Voltammetry, Differential Pulse Voltammetry, Square Wave Voltammetry; Multi-Current Steps; Potentiometric Stripping; RDE/RRDE techniques and many others Devices and Materials Characterizations (e.g. batteries, sensors and biochips, etc.): Charge/Discharge (static and cyclic), Constant Power Tests, Multi-Vertex Scan, Noise Power Spectrum, and on-demand Potentiostatic and Galvanostatic controls with Auto-Stop Criteria (i.e. user-configurable threshold limits on potential, current, charge and power capacity in auto-stopping experiments) Corrosion Techniques: Potentiodynamic Polarization, Galvanostatic Polarization, Linear Polarization Resistance, Potentiodynamic Polarization, Tafel plot, Electrochemical Impedance Spectroscopy, etc. Spectroelectrochemistry: supported via built-in DA/AD interface with trigger control and software batch-communication for precise synchronization with external instruments 	
	DA/AD Signal Acquisitions	Supporting simultaneous multi-channel analog and digital signal-communications with DA or TTL triggers; built-in fast-speed and low-noise DAC; supporting up to 32 user-defined data traces to be recorded and computed.	

Components and Items	Technical Specifications		
	HEKA's SKP method scans a vibrating capacitor probe to image the work function of a surface in air at the micron scale. This method allows simultaneous topography imaging and CPD measurements within one scan. Supported probes are stainless steel, platinum/iridium and tungsten.		
Scanning Kelvin Probe	Probe diameter	10 – 100 µm	
	Surmountable topography changes	50 µm (step size 1.5 nm)	
	Backing Potential	0 – 10 V	
Extendable Peripherals	Supporting online synchronization of a wide range of analytical equipment, such as: RDE/RRDE systems, FTIR spectrometers, UV-Vis Light Source and spectrometers, Photomultiplier tube (PMT), Filter Exchangers, temperature controllers, perfusion system, and research grade CMOS/EMCCD super-resolution cameras.		

Microelectrodes (Pt / Au / Ag / Carbon)

Microelectrode Polishing Machine



Electrochemical Cell Stage Insert with Gas Purging and Temperature Control (5-65°C)



Holders for Microelectrodes, Micropipettes and SMA-Fibers









For over 50 years HEKA has designed and manufactured sophisticated instrumentation and software for biomedical and industrial research applications. Through the years, HEKA has achieved an unparalleled reputation for precision and quality. Medical, pharmaceutical and industrial research facilities world-wide rely on HEKA ingenuity for their discoveries.

While there have been many changes in research, instrumentation, and software, our commitment to bring innovative technology to our customers remains constant. HEKA is a select group of engineers, biomedical researchers, and computer scientists who pride themselves on the quality of HEKA products. HEKA offers complete pre- and post-sales technical support, and takes care of each customer personally. In every way, HEKA provides solutions.



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