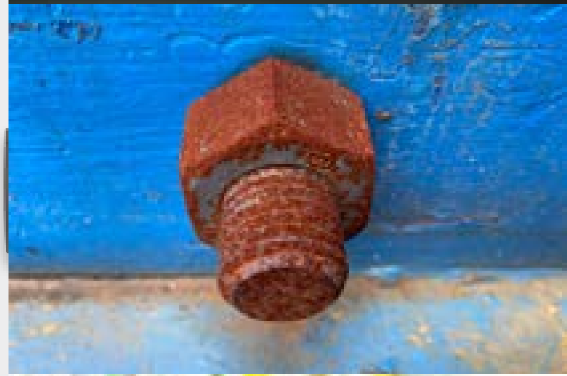
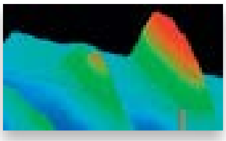
SCANNING-PROBE
SYSTEMS

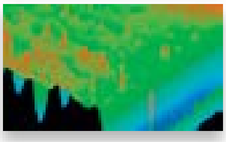
M470.

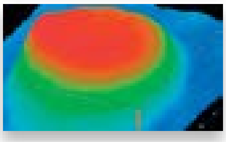
A modular, state-of-the-art instrument allowing users to exploit 9 local electrochemistry techniques

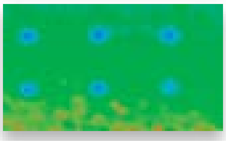


M470 is the 4th generation of scanning probe systems, which includes a high-resolution scanning stage and additional probe techniques.









The M470 achieves the perfect balance of scan speed, resolution and accuracy to deliver a new standard in spatially resolved electrochemical measurements.

Outstanding performance

The fast precise closed loop positioning system is designed specifically for the demands of scanning probe electrochemistry with nanometer resolution. Combined with BioLogic's unique hybrid 32-bit DAC technology, it enables the user to select the configuration most suited to their experiments.

Advanced and flexible platform

The system is available with a combination of nine techniques which make the M470 the world's most flexible scanning probe electrochemistry platform. The M470 can be coupled to BioLogic's Premium range of potentiostats.

Wide range of options

A choice of seven modules, four different cells and a wide range of probes is offered. This is complemented by a long working distance video camera option, glove box cables, and post processing data analysis software.

AVAILABLE TECHNIQUES

- Scanning ElectroChemical Microscopy (SECM)
- alternating current Scanning ElectroChemical Microscopy (ac-SECM)
- intermittent contact Scanning ElectroChemical Microscopy (ic-SECM)
- Localised Electrochemical Impedance Spectroscopy (LEIS)
- Scanning Vibrating Electrode Technique (SVET)
- Scanning Droplet System (SDS)
- alternating current Scanning Droplet System (ac-SDS)
- Scanning Kelvin Probe (SKP)
- Optical Surface Profiler (OSP)

M470 positioning system

Ultra high resolution scanning stage with an extended travel distance

The M470 scanning stage combines high resolution and a large travelling distance as well as a high speed scan.

The scanning system is based on ultra-high precision linear positioning components and offers a high resolution, coupled with a long travel distance.

ic-SECM uses a piezo for fine positioning on the z-axis for topography measurement and relief.

A 20-bit DAC is used for control over the piezo range, and provides position control to 0.09 nm. The M470, with its improved reproducibility and increase in scan speed, can reduce the time taken to run samples.
This lends itself to application areas where the system under study is not in a steady-state.

M470 specifications

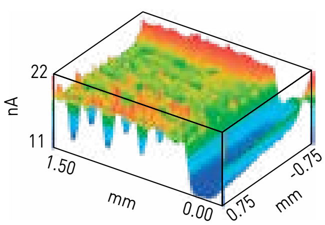
Workstation (all techniques)	
Scan range (x, y, z)	110 mm on all axes
Scan motor resolution	9.76 nm
Closed loop positioning	Linear zero hysteresis encoder with direct real-time readout of displacement in x, y and z
Axis resolution (x, y, z)	20 nm
Max. scan speed	10 mm/s
Measurement resolution	32-bit decoder @ up to 40 MHz
Piezo ic-SECM	
Vibration range	20 nm - 2 µm peak to peak with 1 nm increments
Min. vibration resolution	0.12 nm calculated (16-bit DAC on 4 µm)
Piezo crystal extension	100 µm
Positioning resolution	0.09 nm calculated (20-bit DAC on 100 µm)
Mechanical and electrical	
Scan head	500 x 400 x 675 mm (H x W x D)
Scan control unit	370 x 450 x 420 mm (H x W x D)
Power	250 W

SECM470/ac-SECM470

Local measurement of sample conductivity and electrochemical activity with chemical selectivity

Impedance capability is included as standard for measurement without a redox mediator

Constant distance or **constant height**



SECM is a highly versatile technique which can be applied to a wide range of sample types regardless of conductivity. SECM has been applied to a wide variety of fields from batteries to biology.

The M470 couples to the SP-300 to perform low current, low-noise measurements simultaneously at the tip and substrate, to address specialist applications including those in surface science and the study of living cells.

The M470 includes ac-SECM as a standard experiment type. Using ac-SECM, surface phenomena can be studied without the need for a redox mediator. This feature is applicable in any field where the mediator could adversely affect the reaction under study, including corrosion.

Using the M470, either constant height or constant distance SECM can be performed. Constant distance SECM is achieved by combining SECM470 with the OSP470 or ic-SECM470 module. Experimental data collected using the SECM470 can be imported into the modelling and analysis software package MIRA, giving access to experimental and kinetic parameters.

Typical application areas are:

Biology

- Investigate cell morphology
- Follow photosynthetic processes
- Examine the effects of toxins on biological samples

Catalysis

- High throughput screening of combinatorial libraries of catalysts
- Understand the degradation of Li-Ion electrodes
- Investigate the distribution of materials in an electrode

Coatings & Corrosion

- Investigate the ability of smart coatings to self-heal after damage
- Determine where corrosion is occurring locally
- Follow the formation and breakdown of the passive layer

Photovoltaics

- Investigate sensitizers and electrolytes for dye sensitized solar cells

Sensors

- Study antigen binding in biosensors

SECM470/ac-SECM470 specifications

Potentiostats	
Compliance voltage	±12 V
Applied potential and resolution	±10 V FSR @ 16-bit (down to 1.5 µV)
Measured potential and resolution	± 10 V FSR @ 16-bit (down to 76 µV)
Current ranges	11-decades 100 pA to 1 A
Maximum current	±500 mA
Current resolution	76 aA
Accuracy (+20°C ≤ T ≤+30°C)	< ±0.1% of range ±0.03% of setting for ±500 mA to ±100 nA ranges < ±0.1% of range ±1% of setting for ±10 nA range to ±1 nA ranges < ±0.2% of range ±2% of setting for ±100 pA range
Floating capability	Standard
Cell connections	2, 3 or 4
Scan rate	1 µV/s to 200 V/s
EIS Capability	
Frequency range	10 µHz to 1 MHz
Analyser accuracy	1%, 1°
Max. frequency resolution	0.1 nHz

ic-SECM470

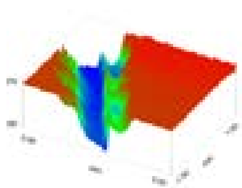
High resolution SECM with simultaneous topography measurement and relief

Intermittent contact SECM allows the user to measure separate topographical and electrochemical information in a single experiment.

Constant distance ac-SECM or dc-SECM

It is well-known that classical SECM measurements are sensitive to surface activity variations as well as topographical changes, therefore it can be beneficial to perform SECM in the constant distance mode.
The solution exploits an innovative tip positioning method to perform constant distance SECM. Through ic-SECM both the surface topography and activity are resolved simultaneously and independently in a single pass experiment. The intermittent contact technique uses the standard SECM probes to follow the topography of the sample throughout the course of a scan.

Using the ic-SECM470 SECM can be performed at the push of a button. The surface approach can be performed automatically allowing the M470 to approach, find, and scan the sample. The ic-SECM470 can be used in the dc or ac-SECM mode, to measure a wide range of sample types. The ic-SECM module is offered exclusively by BioLogic following its introduction by the University of Warwick Electrochemistry and Interfaces Group, and is protected by international patent applications.



Typical application areas are:

Batteries

- Locally compare the conductivity of solid-state electrolyte grains and grain boundaries

Coatings & Corrosion

- Image the breakdown of epoxy coatings
- Study the corrosion of welded materials
- Map hydrogen in metallic alloys

Fuel cells

- Examine the mixing of conductive particles in bipolar plates

Materials

- Investigate the electric conductivity and surface topography of boron doped diamond

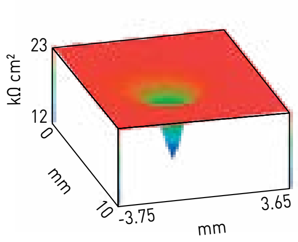
ic-SECM470 specifications

Potentiostats	
Compliance voltage	±12 V
Applied potential and resolution	±10 V FSR @ 16-bit (down to 1.5 µV)
Measured potential and resolution	± 10 V FSR @ 16-bit (down to 76 µV)
Current ranges	11 decades 100 pA to 1 A
Maximum current	±500 mA
Current resolution	76 aA
Accuracy	< ±0.1% of range ±0.03% of setting for ±500 mA to ±100 nA ranges < ±0.1% of range ±1% of setting for ±10 nA range to ±1 nA ranges < ±0.2% of range ±2% of setting for ±100 pA range
Floating capability	Standard
Cell connections	2, 3 or 4
EIS Capability	
Frequency range	10 µHz to 1 MHz
Analyzer accuracy	0.1 nHz
ic-SECM module	
Tip control	Piezo element and stepper
Piezo crystal extension	100 µm
Vibration frequency	80 - 600 Hz
Vibration control	20 nm - 2 µm peak to peak
Minimum increment	1 nm
Z control resolution	0.09 nm (piezo)
Topology resolution (recorded)	1 µm
Positioning -probe to sample surface	Autonomous

LEIS470

Localised impedance measurements

Measurements can be made using the powerful inbuilt sequencer to perform local frequency sweeps at set points across a surface, or map a surface switching frequencies for each map.



The principles of Localised Electrochemical Impedance Spectroscopy (LEIS) are similar to those employed in EIS, in that a small sinusoidal voltage perturbation is applied to a working electrode sample and the resulting current is measured to allow the calculation of the impedance.However, rather than measuring the bulk current, a small electrochemical probe is scanned close to the surface, measuring the localised current in the electrolyte.

Producing area maps over a sample at a single frequency has never been easier. To easily investigate changes to the sample over time the software sequencer can be used to loop LEIS area scan measurements. The LEIS470 also allows the user to make galvanic or global impedance measurements simply by choosing which mode the potentiostat is operating in, and the software does the rest.

Typical application areas are:

Batteries

- Explore the effect of surface treatments on the electrodes

Coatings

- Study the ability of smart coatings to self-heal after damage
- Investigate the occurrence of underfilm corrosion
- Follow coating failure and delamination

Corrosion

- Examine the effect of alloy composition on corrosion
- Study pit initiation of corrosion
- Investigate the corrosion resistance of metal hydrides used in hydrogen storage

LEIS470 specifications

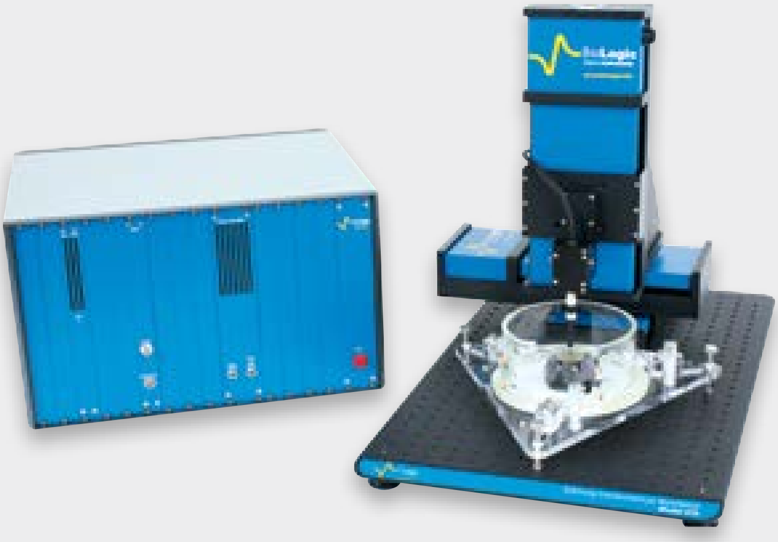
Potentiostat	
Compliance voltage	±12 V
Applied potential and resolution	±10 V FSR @ 16-bit (down to 1.5 µV)
Measured potential and resolution	± 10 V FSR @ 16-bit (down to 76 µV)
Current ranges	11-decades 100 pA to 1 A
Maximum current	±500
Current resolution	76 aA
Accuracy (+20°C ≤ T ≤+30°C)	< ±0.1% of range ±0.03% of setting for ±500 mA to ±100 nA ranges < ±0.1% of range ±1% of setting for ±10 nA range to ±1 nA ranges < ±0.2% of range ±2% of setting for ±100 pA range
Floating capability	Standard
Cell connections	2, 3 or 4
Scan rate	1 µV/s to 200 V/s
EIS capability	
Frequency range	10 µHz to 3 MHz
Analyser accuracy	1%, 0.1
Max. frequency resolution	0.1 nHz

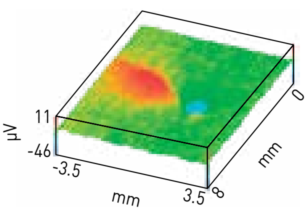
SVP470

In situ measurement of localised electrochemical activity

The Scanning Vibrating Electrode Technique (SVET) maps the electric field generated in a plane above the surface of an electrochemically active sample.

Signal extraction: user-tuned or auto-tuned





SVET enables the user to map and quantify localised electrochemical events in real time. As a result SVET is highly applicable to corrosion studies, and is used in biology, where it is known as Vibrating Probe, to investigate extracellular currents.

The probe vibration is controlled by a piezoelectric vibration actuator allowing vibration amplitudes from 1 - 30 µm (perpendicular to the sample surface). It is an ac technique, thus, high system sensitivity can be achieved via a differential electrometer in conjunction with a lock-in amplifier.

The SVP470 vibrating probe provides increased electrical sensitivity as well as enhanced system stability.

By choosing a BioLogic potentiostat, users can access an integrated suite of dc corrosion experiments and can be used to bias the potential of the sample.

Typical application areas are:

Batteries

- Follow the effect of changing the applied potential on the activity of aqueous battery electrodes

Biology

- Follow photosynthetic processes
- Map the electric fields associated with wound healing

Coatings

- Investigate the ability of smart coatings to self-heal after damage
- Image the cathodic protection of coatings

Corrosion

- Investigate the effect of sample structure on corrosion
- Follow the electrochemical progress of corrosion in real time
- Study galvanic corrosion *in-situ* in real time

SVP470 specifications

SVP electronics	
Signal chain	Phase sensitive detection using microprocessor controlled lock-in amplifier with digital dual phase oscillator and differential electrometer input
Lock-in amplifier	Software controllable gain range (1 - 10^5). Output time constant 0.01, 0.1, 1, 10 s
Differential electrometer	10^{15} Ohms input impedance. Decade gain ranges 0 to 80 dB. Common mode range ±12 V
Vibration actuator	One dimensional low voltage piezoelectric actuator
Vibration amplitude	Software set from 1 - 30 µm perpendicular to sample surface
Electrochemical sensitivity	Better than 5 µA/cm^2 (using standard PIS test approach)

SKP470

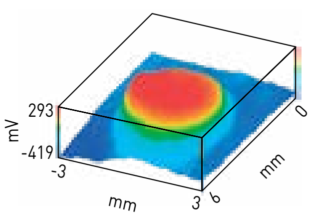
Surface potential and topography measurements

Scanning Kelvin Probe (SKP) is a non-contact, non-destructive technique which is highly sensitive to changes in work function due to changes in the surface state.

Signal extraction: user-tuned or auto-tuned

Using a vibrating capacitance probe, the SKP470 Scanning Kelvin Probe measures the work function difference between the scanned probe reference tip and sample surface. The measured work function can be directly correlated to many aspects of the surface condition, including corrosion potential.

The use of a capacitance probe allows the SKP470 to measure the sample topography. Through these topography measurements, the SKP470 can be used in height tracking mode to maintain a constant height between the probe tip and sample surface, allowing SKP measurements to be made over uneven surfaces.



Typical application areas are:

Photovoltaics

- Determine the work function of photovoltaic materials

Catalysis

- Measure changes in work function related to the effect of sample treatment on catalytic activity

Coatings & Corrosion

- Study the ability of smart coatings to self-heal after damage
- Image the breakdown of epoxy coatings
- Investigate the effect of alloy composition on corrosion
- Determine where corrosion is occurring locally

Materials

- Measure the effects of dopants on the electrical properties of a semiconductor

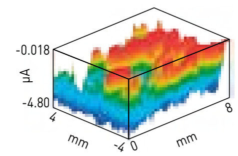
SKP470 specifications

SKP electronics	
Signal chain	Phase sensitive detection using microprocessor controlled lock-in amplifier with digital dual phase oscillator and differential electrometer input
Lock-in amplifier	Software controllable gain range (1 – 10^5). Output time constant 0.01, 0.1, 1, 10 s
Differential electrometer	10^{15} Ohms input impedance. Decade gain ranges 0 to 80 dB. (1x to 10000x). Common mode range ±12 V
Vibration actuator	One dimensional low voltage piezoelectric actuator
Vibration amplitude	Software set from 1 - 30 µm perpendicular to sample surface
Backing Potential Controller (BPC) potential range	±10 V
BPC DAC Resolution	300 µV
BPC sampling	0.1 Hz to 1000 Hz
BPC type	PID controller
Probe	
Probe type	Proprietary SKPR tungsten air gap. Available in 150 and 500 µm diameter

SDS470/ac-SDS470

Voltammetry and impedance measurements at the micrometric scale

The second generation of the Scanning Droplet System incorporates a small aperture, precision machined reservoir type SDS head with a significant increase in the resolution of electrochemical events in the microcell.



The Scanning Droplet System uses a Scanning Droplet Cell (SDC) that allows a spatially resolved, *in situ* investigation by all standard electrochemical techniques. SDC is a technique which confines a liquid in contact with a sample surface in order to measure electrochemical and corrosion reactions over a limited region where the droplet is actually in contact with the sample.

This offers the unique ability to spatially resolve electrochemical activity and to confine it exclusively to a quantifiable area of the sample.

The scanning droplet system allows the positioning of a small drop of electrolyte from the SDS head onto the sample surface. The wetted surface area under investigation acts as the working electrode and the SDS head contains the counter and reference electrodes which are electrically connected to the surface through the drop. The SDS470 is also supplied with a flow type SDS head for studies in which continuous regeneration of the droplet are key.

Typical application areas are:

Catalysis

- High throughput screening of combinatorial libraries of catalysts

Corrosion

- Probe the effect of features, like welds, on corrosion properties
- Investigate the effect of flow on corrosion
- High throughput screening of corrosion properties of alloy libraries

Materials

- High throughput screening for material discovery and optimization
- Investigate coarse grain crystalline materials

Photovoltaics

- Study the local effect of doping on donor/acceptor materials

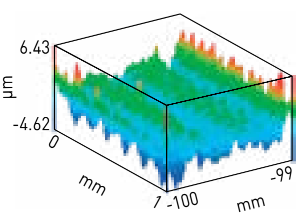
SDS470/ac-SDS470 specifications

SDS Head	
Included SDS Heads	500 µm PEEK, 100 µm MACOR®
500 µm Flow Head	
Type	Flow
Reference Electrode	Ag/AgCl within sensor head
Counter Electrode	Pt wire within capillary
Head Construction	PEEK with silicone rubber tubing
Aperture	ID 500 µm, 0.196 mm^2
Resolution	< 1 mm depending on solution/ surface
100 µm Reservoir Head	
Type	Reservoir
Reference Electrode	Pt wire
Counter Electrode	Pt wire
Head Construction	MACOR® Glass Ceramic
Aperture	ID 100 µm, 0.00785 mm^2
Resolution	200 µm depending on solution/ surface
Pump System	
Type	Peristaltic
Channels	2-Channels
Potentiostat	
Compliance voltage	±12 V
Applied potential and resolution	±10 V FSR @ 16-bit (down to 1.5 µV)
Measured potential and resolution	± 10 V FSR @ 16-bit (down to 76 µV)
Current ranges	11-decades 100 pA to 1 A
Maximum current	±500 mA
Current resolution	76 aA
Accuracy (+20°C ≤ T ≤+30°C)	< ±0.1% of range ±0.03% of setting for ±500 mA to ±100 nA ranges < ±0.1% of range ±1% of setting for ±10 nA range to ±1 nA ranges < ±0.2% of range ±2% of setting for ±100 pA range
Floating capability	Standard
Cell connections	2, 3 or 4
Scan rate	1 µV/s to 200 V/s
EIS capability	
Frequency range	1 0 µHz to 1 MHz
Analyser accuracy	1%, 1°
Max. frequency resolution	0.1 nHz

OSP470

Non-contact topography measurements

Utilising a non-contact laser displacement sensor, the OSP470 module allows fast and accurate non-contact surface measurement to a very high accuracy.



Features with a step height of less than 1 µm can be imaged and measured over a height measurement range of 10 mm without touching the sample surface. The OSP470 incorporates a CCD displacement sensor mounted on the scanning head of the M470 workstation. A tightly focused laser (650 nm) is projected onto the sample surface and the scattered light is detected by a CCD array allowing the direct displacement measurement of the diffuse scattered light. This allows a very accurate surface height profile of the entire surface to be generated and allows measurements of the surface roughness and topography features.

Most importantly, the OSP470 module will allow the use of topography data to alter the height of the probe in many of the other electrochemistry techniques. The probe is then able to scan over uneven surfaces whilst maintaining a constant distance of the probe from the sample.

Typical application areas are:

Coatings

- Examine the morphology of polymer materials
- Study the surface roughness of aerospace coatings
- Investigate damage to coated metal surfaces

Corrosion

- Measure sample topography for height tracking corrosion investigations
- Investigate changes in topography due to corrosion attacks
- Examine topography changes due to biofilm formation

Materials

- Investigate the effects of water absorption on material swelling

Biology

- Investigate the effect of surface texture on barnacle settlement

OSP470 specifications

Sensor	
Measurement range	10 mm
Reference distance	30 mm
Maximum vertical resolution (static)	100 nm
Spot size	30 µm at focus
Scan speed	10 mm/s
Multiple readings averaged	Yes
Correct positioning	Red light/green light
Light source	650 nm class 2 semiconductor laser max. 0.95 mW
Auto-calibration for off-axis alignments	Yes
Real-time CCD readout	Yes

Accessories

A wide range of accessories, specifically developed for scanning probe techniques, is available for the M470 system

Shallow Tricell™

Environmental Tricell™

Cells

The µTriCell™ range of electrochemical cells for the M470 are ideal for SECM experiments. They require a low volume of electrolyte, while allowing users to easily adjust the sample position. The µTriCell™ (not shown) can accommodate a variety of sample configurations while maintaining easy electrical connection. The Shallow µTriCell™ has been designed for constant-distance scanning probe experiments. It can accommodate a variety of sample configurations while allowing wide optical access. The Foil Cell accessory (not shown) in the µTriCell™ range has been designed for SECM studies of flat foil sample, particularly those used as battery electrodes.

The Environmental TriCell™ system with its four inlet/outlet purge ports and an optional rubber gaiter cover seal is ideal for controlled atmosphere, liquid flow or temperature control experiments.

Glove Box Cable Sets

A range of glove box cable sets are available to replace the standard M470 cables for use of the M470 in a glove box. The available cable sets are supplied with the necessary feedthroughs to allow connection.

USB-PIO

The USB-PIO is an optional accessory for use with the M470.
The USB-PIO is designed to switch external devices from within the M470 software.

Video Imaging System

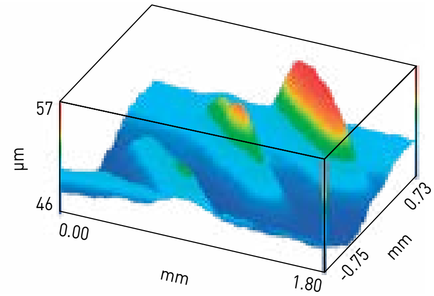
The Video Microscope System (model VCAM3) is a long working distance video microscope which allows users to view the distance between probe tip and sample surface in many scanning probe electrochemistry techniques.

Probes

A range of probes dedicated for use with our SVP, SKP and LEIS scanning probe applications are available for the M470 system.

A range of ultra-microelectrodes (1-25 µm diameter) is available for the SECM470 system.

They have been designed to ensure robustness in everyday use with a manufacturing process that closely controls the diameter of glass at the probe tip.



Software

Complete control and analysis tools

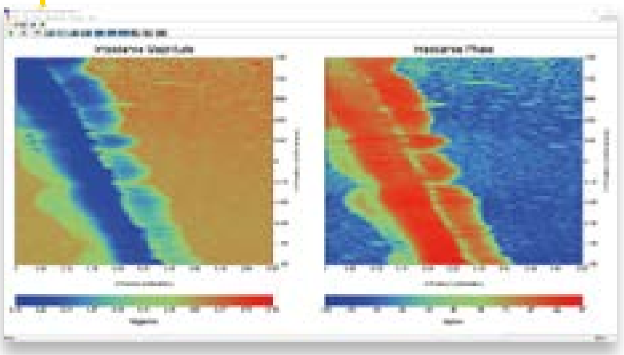
The M470 software supports all the available techniques and uses a standard experiment model. The optional 3DIsoPlot™ and MIRA software provide extended analysis and imaging features. The software automatically recognises the installed techniques and seamlessly incorporates any experiment specific parameters.

The instrument is configured for area and line experiments and incorporates standard dc and ac techniques. It also allows the user to easily define, visualise, record and configure all experiment parameters as well as analyse and manipulate data post-experiment.

M470

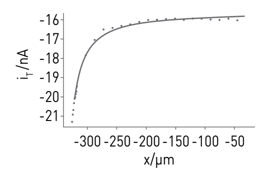
The M470 software includes:

- SECM automated approach curve, with user-definable approach curve step size change and termination
- Higher resolution readout
- Manual or automatic tuning of the demodulation phase used in SVP and SKP
- Area map post processing including tilt correction, X or Y curve subtraction and 2D and 3D FFT
- Auto-sequencing of experiments, probe movement and area mapping
- Graphical Experiment Sequencing Engine (GESE)
- Support for multi-zone scanning
- Multiple data views for all experiments
- Peak analysis



3DIsoPlot™

3DIsoPlot™ features a user friendly interface and is ideal for displaying 3D maps of data produced by our range of scanning probe electrochemistry systems. 3DIsoPlot™ is suitable for displaying a wide range of data types from scanned to mathematical modelling data. 3DIsoPlot™ produces 3D plots in the form of shaded surfaces. Wire frame plots and colour contour maps of the surface are also available.



MIRA

MIRA (Microscopic Image Rapid Analysis) is an extremely powerful tool for the representation and analysis of data obtained by any scanning probe microscopy technique. It features an extensive range of 2D and 3D data representation tools for area scan data obtained with SECM. The package also has the ability to fit approach curve data using a wide range of equations which correspond to the conditions of the approach curve: with or without current offset, approach to a conductor or an insulator, generation/collection mode, etc. Such fitting gives access to parameters such as the actual probe to sample distance, the RG factor, the tip current in semi-infinite condition, the tip radius, amongst many others.