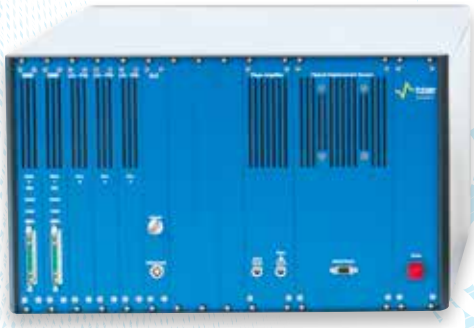
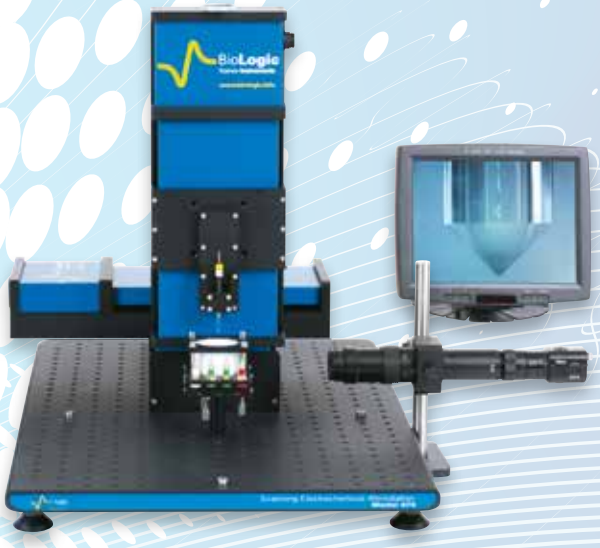
M470

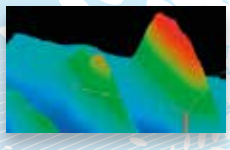
Scanning probe electrochemistry with high resolution and high modularity

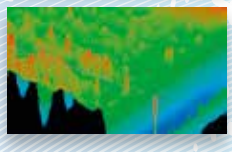


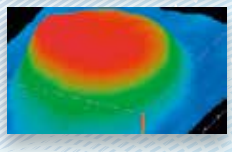


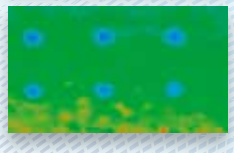
BioLogic

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 Uniscan'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 470 the world's most flexible scanning probe electrochemistry platform.

Wide range of options

A choice of seven modules, three different cells, wide range of probes, long working distance video camera option, post processing data analysis software options is offered.

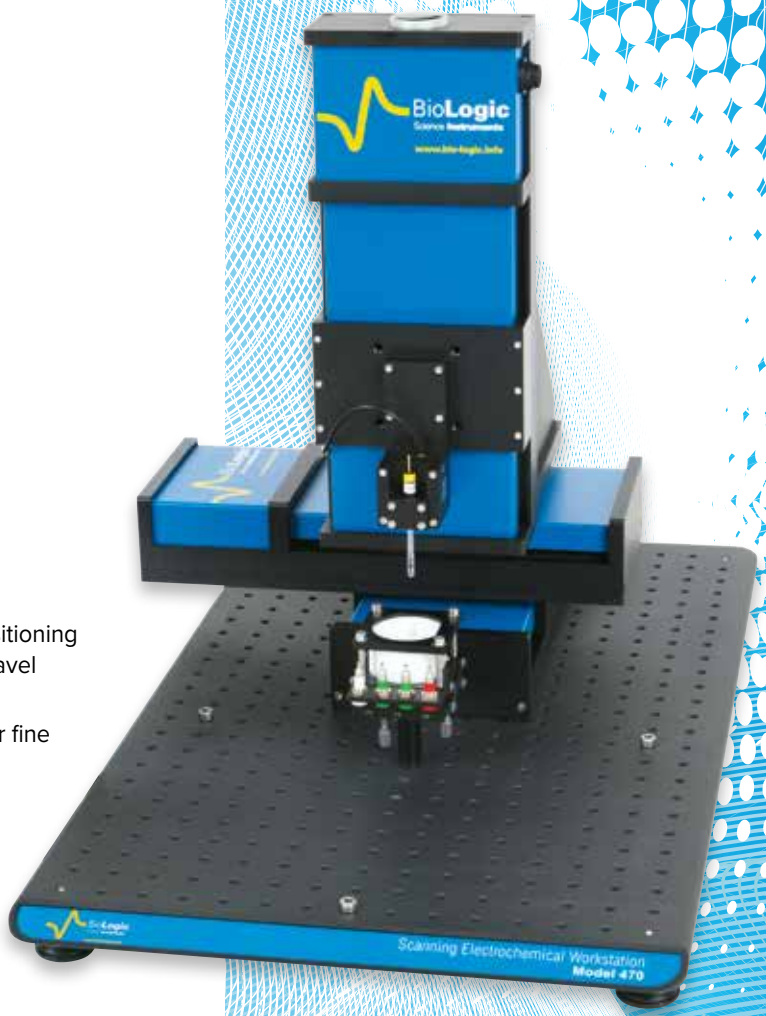
AVAILABLE TECHNIQUES

- 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 new M470 scanning stage combines an increased resolution, an increased 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.

Some of the newer techniques (ic- and ac-) also offer a piezo for fine positioning on the z-axis for topology 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 steady-state.

M470 specifications

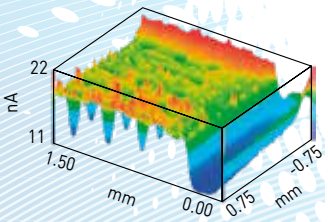
Workstation (all techniques)	
Scan range (x, y, z)	Greater than 100 mm
Scan motor resolution	9.76 nm
Closed loop positioning	Linear zero hysteresis encoder with direct real-time readout of displacement in x, y and z
Axis resolution (x, y, z)	20 nm
Max. scan speed	10 mm/s
Measurement resolution	32-bit decoder @ up to 40 MHz
Piezo (ic- and ac-techniques)	
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 420 x 675 mm (H x W x D)
Scan control unit	275 x 450 x 400 mm (H x W x D)
Power	250 W

SECM470/ac-SECM470

Constant distance or **constant height**

Measurement of the surface conductivity and reactivity with and without a mediator

The Scanning ElectroChemical Microscope is now available with impedance capability.



The 3300 bi-potentiostat/FRA can make low current, low noise measurements simultaneously at the tip and substrate to address specialist applications in surface science and the study of living cells.

ac-SECM is a new technique that has the capability to measure surface phenomena without the presence of a mediator. This feature is useful in the field of corrosion as it allows measurement without the need for additional chemicals.

The SECM technique can be combined with the OSP470 module or the ic-SECM470 module and make both constant height and constant distance measurements.

The SECM470 has a new stage for the SECM probe, which holds the electrode tight and parallel to the Z-axis.

Experimental data can be imported into the modelling and analysis software package MIRA, giving access to experimental and kinetic parameters.

Typical application areas are:

Materials

- Investigation of soft biological material
- Design of functional materials
- Fabricated surfaces for sensors
- Characterisation of combinatorial libraries for fuel cell applications

Fundamentals

- Corrosion mechanisms
- Ion fluxes through pores
- Investigating cellular activity of living cells
- SECM imaging in the absence of a redox active species

Corrosion

- Corrosion activity of polymer defects
- Surface phenomena in the absence of a mediator
- Characterisation of metallic surfaces with ac-SECM

Generated data at each point:

- ac current and voltage magnitude and phase
- dc probe and sample measurement
- Impedance magnitude and phase

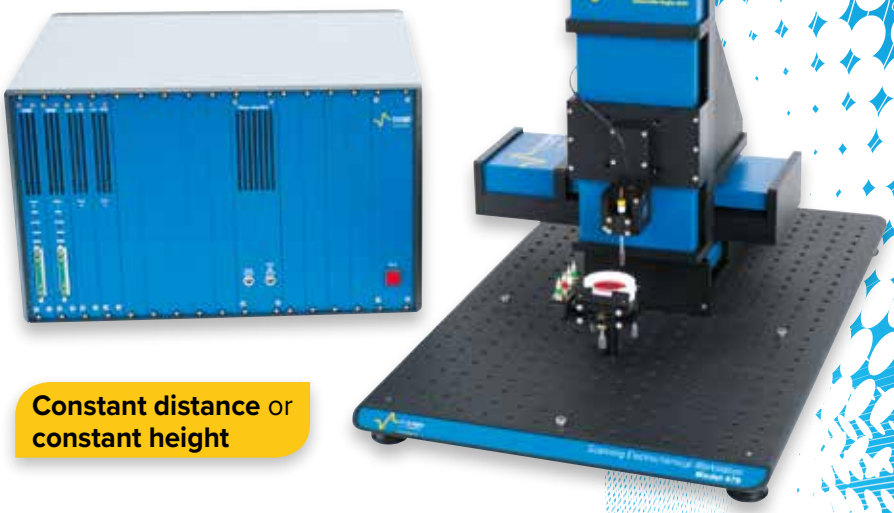
SECM470/ac-SECM470 specifications

Potentiostats	
Compliance voltage	±12 V
Applied potential and resolution	±10 V FSR @ 32-bit (4.7 nV)
Measured potential and resolution	±10 V FSR @ 24-bit (1.2 µV)
Current ranges	10-decades 1 nA to 1 A
Maximum current	500 mA
Current resolution	23.8 fA
Accuracy	<0.5%
Floating capability	Standard
Cell connections	2, 3 or 4
Maximum ADC sample rate	4 MHz
Maximum ADC resolution	24-bit
Minimum pulse duration	100 µs
Scan rate	1 µV/s to 200 V/s
Electrometer	
Impedance	10^{13} Ω II 7 pF typical
Bias current	1 pA typical
Bandwidth	1 MHz
EIS capability	
Frequency range	1 µHz to 1 MHz
Analyser accuracy	0.1%, 0.1°
Max. frequency resolution	66 nHz

ic-SECM470

High resolution SECM with simultaneous topography measurement and relief

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



Constant distance or **constant height**

It is well-known that classical SECM measurements are sensitive to surface conductivity variations as well as topographical changes.

ic-SECM470 introduces a brand new technique that allows surface topography and activity to be resolved simultaneously and independently by using an innovative tip positioning method.

The intermittent contact technique allows an SECM probe to follow the topography of the sample over the course of a scan. It uses standard SECM probes.

An approach curve for an area scan experiment can be performed automatically or as directed by the user.

Press a button and the M470 approaches, finds and scans the surface.

The ic-SECM module has been developed by Uniscan Instruments following its recent introduction by the University of Warwick Electrochemistry and Interfaces Group, and is protected by international patent applications.

Typical application areas are:

Materials

- Visualization of molecular transport through semi permeable membranes

Fundamentals

- Simultaneous measurement of surface topography and electrochemistry
- Assessing electrochemical microcell geometry
- Investigations into the electric conductivity and surface topography of Boron doped diamond

Corrosion

- Delaminated areas beneath organic coating
- Corrosion of welded material
- Localised corrosion in oilfield formation waters

Generated data at each point:

- Topology
- dc probe and sample measurement
- Probe ac magnitude and phase

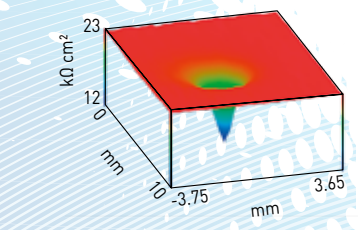
ic-SECM470 specifications

Potentiostats	
Compliance voltage	±12 V
Applied potential and resolution	±10 V FSR @ 32-bit (4.7 nV)
Measured potential and resolution	±10 V FSR @ 24-bit (1.2 µV)
Current ranges	10-decades 1 nA to 1 A
Maximum current	500 mA
Current resolution	23.8 fA
Accuracy	<0.5%
Floating capability	Standard
Cell connections	2, 3 or 4
Maximum ADC sample rate	4 MHz
Maximum ADC resolution	24-bit
Minimum pulse duration	100 µs
Scan rate	1 µV/s to 200 V/s
Electrometer	
Impedance	10^{13} Ω ll 7 pF typical
Bias current	1 pA typical
Bandwidth	1 MHz
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 for step or sweep modes

LEIS470

Localised impedance measurements

They are made using the powerful inbuilt sequencer to perform frequency sweeps across a surface or map a surface and switch frequencies for each measurement.



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. 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:

Materials

- Plastic deformation and microstructures
- Ageing and shock processing

Fundamentals

- Scanning photo-induced impedance microscopy
- Assessing electrochemical microcell geometry

Corrosion

- Delaminated areas beneath organic coating
- "SMART" protection of water based epoxy coatings
- Single pit initiation on 316L stainless steel
- Localised corrosion in oilfield formation water

LEIS470 specifications

Potentiostat	
Compliance voltage	±12 V
Applied potential and resolution	±10 V FSR @ 32-bit (4.7 nV)
Measured potential and resolution	±10 V FSR @ 24-bit (1.2 µV)
Current ranges	10-decades 1 nA to 1 A
Maximum current	500 mA
Current resolution	23.8 fA
Accuracy	<0.5%
Floating capability	Standard
Cell connections	2, 3 or 4
Maximum ADC sample rate	4 MHz
Maximum ADC resolution	24-bit
Minimum pulse duration	100 µs
Scan rate	1 µV/s to 200 V/s
Electrometer	
Impedance	10^{13} Ω II 7 pF typical
Bias current	1 pA typical
Bandwidth	100 kHz
EIS capability	
Frequency range	1 µHz to 1 MHz
Analyser accuracy	0.1%, 0.1°
Max. frequency resolution	66 nHz

SVP470

In situ measurement of the 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.

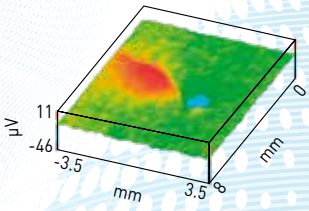
Signal extraction:
user-tuned or **auto-tuned**

This enables the user to map and quantify localised electrochemical and corrosion events in real time.

The probe vibration is controlled by a piezo-ceramic displacement device 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.

The 3300 potentiostat is an option that offers a fully integrated suite of dc corrosion experiments and can be used to bias the potential of the sample.



Typical application areas are:

Materials

- Hybrid coatings on steel
- Galvanic corrosion of an iron–zinc cell
- Electroactive conducting polymers (ECPs) for corrosion-resistant coatings

Fundamentals

- Studies of the multifunctional molecular layer between metallic Zn and polymer coatings on Fe
- *In situ* characterization of the electrochemistry of grain boundaries

Corrosion

- Imaging of the cathodic protection of coatings
- Quantitative assessment of localised dissolution of stainless steels
- Use of amino alcohol based inhibitors to control corrosion of reinforcing steel

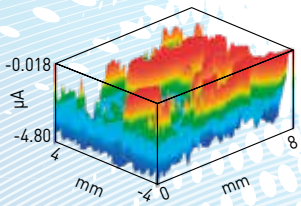
SVP470 specifications

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 0.1, 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 piezo-electric 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)

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 MACOR® Glass Ceramic scan 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. SDS 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 technique allows the positioning of a small drop of electrolyte from a 100 µm capillary onto the sample surface. The wetted surface area under investigation acts as the working electrode and the capillary contains the counter and reference electrodes which are electrically connected to the surface through the drop.

Typical application areas are:

Materials

- High throughput screening for material discovery and optimization
- High throughput characterisation of combinatorial material libraries

Fundamentals

- Investigation of structured oxide films
- Localised investigation of coarse grain crystalline materials
- *In situ* study of repassivation

Corrosion

- Corrosion-potential profiles of brazing sheets
- Investigations of single grain polycrystalline materials and their grain boundaries

Generated data at each point:

- ac current and voltage magnitude and phase
- dc sample measurement
- Impedance magnitude and phase

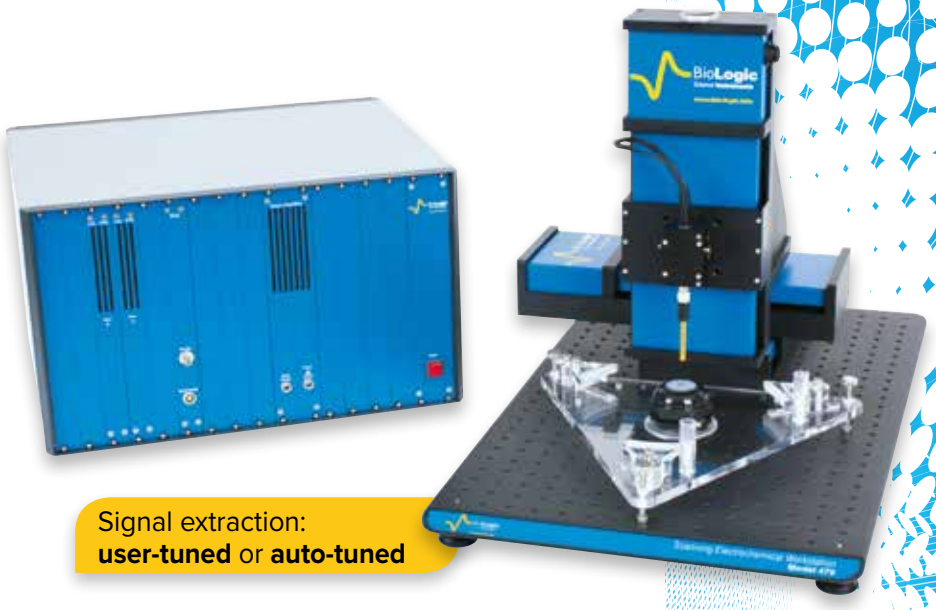
SDS470/ac-SDS470 specifications

SDS head	
Reference electrode	Ag/AgCl within sensor head
Counter electrode	Pt wire inside capillary
Construction material	MACOR® glass ceramic
Aperture diameter	100 µm/0.00785 mm^2
Hole aspect ratio (HAR)	10
Resolution	200 µm depending on solution/surface
Potentiostat	
Compliance voltage	±12 V
Applied potential and resolution	±10 V FSR at 32-bit (4.7 nV)
Measured potential and resolution	±10 V FSR at 24-bit (1.2 µV)
Current ranges	10-decades 1 nA to 1 A
Maximum current	500 mA
Current resolution	23.8 fA
Accuracy	<0.5%
Floating capability	Standard
Cell connections	2, 3 or 4
Maximum ADC sample rate	4 MHz
Maximum ADC resolution	24-bit
Minimum pulse duration	100 µs
Scan rate	1 µV/s to 200 V/s
EIS capability	
Frequency range	1 µHz to 1 MHz
Analyser accuracy	0.1%, 0.1°
Max. frequency resolution	66 nHz

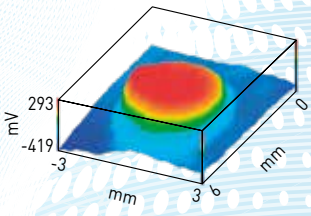
SKP470

Surface potential and topography measurements

Using a vibrating capacitance probe the SKP470 Scanning Kelvin Probe measures the work function difference between the scanned probe reference tip and sample surface.



Signal extraction:
user-tuned or **auto-tuned**



The surface work function can be directly correlated to many aspects of surface condition including corrosion potential. Moreover, as the technique utilises a capacitance probe, it can be used to measure the topography of the sample.

Extension of this allows the technique to be used in height tracking mode to maintain a constant height between the probe tip and sample surface, allowing measurements to be made over uneven surfaces.

The SKP470 is the very latest development in Kelvin probe technology incorporating many new features affording the user performance, flexibility and ease of use.

Typical application areas are:

Materials

- Detection of defects in steel cord - rubber coating interface
- Investigations of sol-gel coatings for surface modification

Fundamentals

- Surface photovoltage analysis of multicrystalline silicon
- Electrochemical studies of iron meteorites

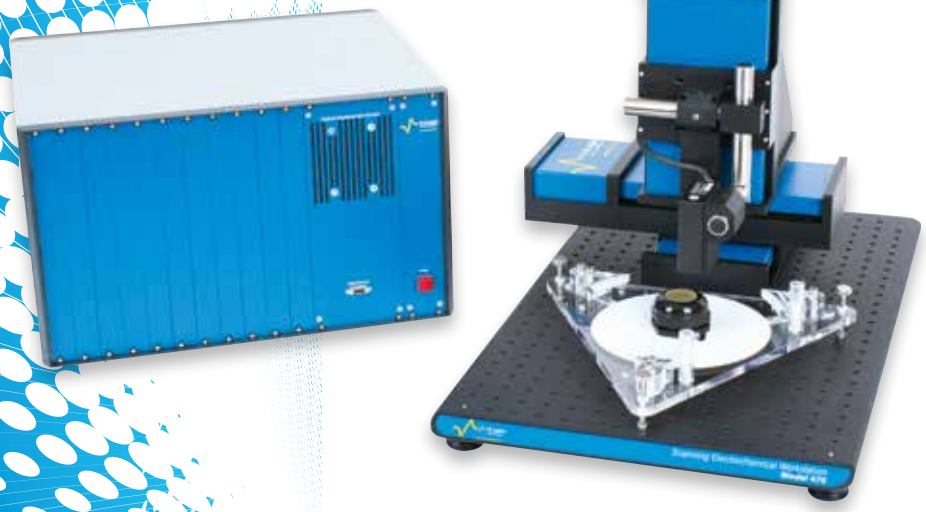
Corrosion

- Studies of alloy-specific corrosion inhibition by novel DOPA containing proteins
- Height regulated studies of polymer-metal interfaces in corrosive environments
- Investigation of filiform corrosion mechanisms

SKP470 specifications

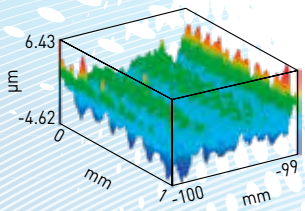
SKP electronics	
Signal chain	Phase sensitive detection using microprocessor controlled lock-in amplifier with digital dual phase oscillator and differential electrometer input
Lock-in amplifier	Software controllable gain range (1 - 10^5). Output time constant 0.1, 1, 10 s
Differential electrometer	10^{15} Ohms input impedance. Decade gain ranges 0 to 80 dB. Common mode range ±12 V
Vibration actuator	One dimensional low voltage piezo-electric 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 1,000 Hz
BPC type	PID controller
Probe	
Probe type	Proprietary SKPR tungsten air gap

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 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 the 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:

Materials

- Surface roughness parameter measurement
- Assessment of vitrified CBN wheels for precision grinding

Fundamentals

- Generation of height profile data to maintain a constant distance of the probe from the sample
- General surface profile measurement of engineering samples

Corrosion

- Surface topography measurements on corroded stainless steel samples
- Coating thickness and smoothness measurement
- Investigations of damage on coated metal surfaces
- Bubbling and delamination

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 developed specifically for scanning probe techniques are available for the M470 system

Cells

The µTriCell™ allows users to adjust the position of samples without moving or adjusting the scanning head and reduces the volume of electrolyte required for scanning probe electrochemistry experiments.

The Shallow µTriCell™ has been designed for constant-distance scanning probe systems and incorporates an extremely flexible sample accommodation platform, designed to take various sample configurations with wide optical access.

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.

Video Imaging System (optional)

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 (10, 15 or 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. 3DIsoPlot™ and MIRA 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.

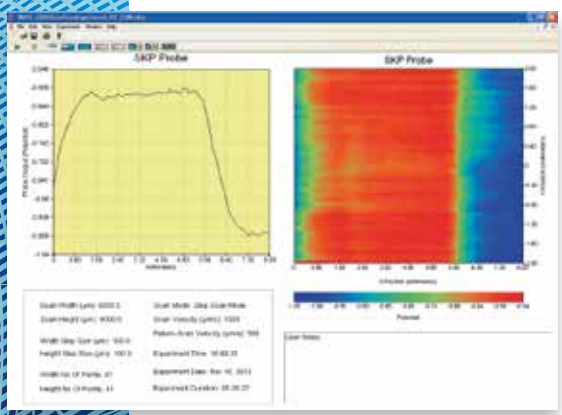
M470

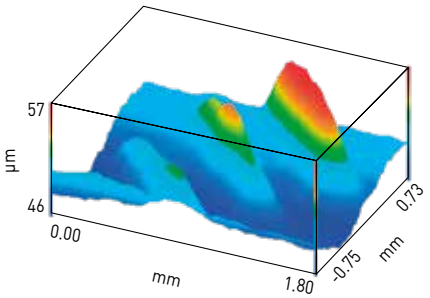
New features are:

- SECM automated approach curve,
- SECM user-definable approach curve step size change,
- higher resolution readout,
- manual or automatic tuning of the demodulation phase.

It also incorporates the following features:

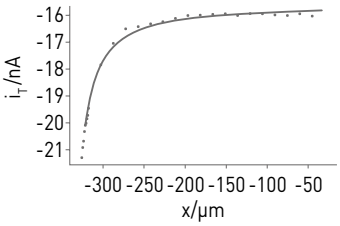
- tilt correction,
- X or Y curve substraction (polynomial up to 5th order),
- 2D and 3D FFT,
- autosequencing of experiments, probe movement and area mapping,
- Graphical Experiment Sequencing Engine (GESE),
- support for multi-zone scanning,
- multiple data views for all experiments,
- peak analysis.





3DIsoPlot™ (optional)

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 (optional)

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 curves 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.

Product designed and manufactured by Uniscan Instruments Ltd, a Bio-Logic SAS company

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