M370

Scanning electrochemical workstation



M370 versatility and precision







The M370 is available in any or all of the six configurations (SECM, SVET, SKP, LEIS, SDS, OSP) and

may be upgraded at a later date by subsequent purchase of any combination of the available techniques. A wide variety of optional accessories are also available, including various probe options, cell options

[Environmental TriCell[™] and µTriCell[™]), long working distance optical video microscope (VCAM3) and 3D shaded surface rendering software (3DIsoPlot[™]).



AVAILABLE TECHNIQUES

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

APPLICATIONS

- Monitoring biological activity
- Determining kinetic parameters
- Imaging immobilized enzymes
- Materials for fuel cell research
 - Batteries
 - Surface modification
- Corrosion science
- Film/coating research
- Surface contaminations
- Sensors
- Photo voltage spectroscopy
- Imaging complex impedance of thin films
- Direct imaging of cell growth media
- Characterisation of photoelectrochemical reactions

M370 positioning system

Long travel distance and high resolution

Zero hysteresis and closed loop positioning ensures submicron resolution.

The M370 utilises a fast and precise closed loop x, y, z positioning system with submicron resolution, along with a flexible data acquisition system enabling the user to select the configuration most suited to their experiments.

The system is designed with flexibility in mind and the design ergonomics ensure convenient cell, sample and probe access.

The ability to configure to a specific application and upgrade at a later date makes the M370 uniquely flexible, whilst maintaining ultimate performance.



 E_{corr} changes due to flow rate variation on mild steel sample (obtained by SDS370).

70 x 70 x 70 mm
8 nm
Yes
100 nm
2 mm/s
16-bit @ 100 kHz
Optical table standard screw fitting
470 x 350 x 660 mm (H x W x D)
140 x 450 x 330 mm (H x W x D)
140 x 450 x 330 mm (H x W x D)

SECM370



SECM image in tip generation/ substrate collection mode of gold on silicon ultra micro-electrode array.

Measurement of the surface conductivity and reactivity

The Scanning ElectroChemical Microscope SECM370 is a precision scanning ultra micro-electrode system which can be used to monitor (or impose) current flowing between an ultra micro-electrode and a specimen surface in solution at extremely high spatial resolutions.

It can be used to examine, analyse or alter the surface chemistry of a sample in solution.

This equipment has many potential applications in the study of fundamentals of surface reactions in fields as diverse as corrosion science to enzyme stability studies.



SECM approach curve to conducting surface for 10 µm diameter probe.

SECM370 specifications

Floating electrochemical cell, all	ows connection in bipotentiostat
Current ranges	8 decades: 1 nA to 10 mA/V
Current resolution	61 fA
Maximum current	±20 mA
Applied probe potential	±2 V @ 16-bit (61 μV res). Factory option to ±8 V*
Applied substrate potential	±2 V @ 16-bit (61 μV res). Factory option to ±8 V*
Compliance voltage	±8 V
ADC resolution	16-bit @ > 100 kHz
Rise time	1 V/μs into 1 kΩ
Current measurement accuracy	<0.5%
I/E input bias	<10 pA

* Resolution will vary with option specified.





Localised impedance map of intact coating on mild steel with sub-film chloride contamination.

Localised impedance measurements

The Localised Electrochemical Impedance Spectroscopy LEIS370 allows spatially resolved impedance measurements to be made, combining established principles of EIS measurements with scanning probe technology.

The principles of 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 local current in the electrolyte.

The LEIS system can be provided with the Uniscan 3300 FRA. Please ask us for a list of other compatible instruments.



Crossview of the LEIS dual electrode.

LEIS370 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/V
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

SVET370

In situ measurement of the localised electrochemical activity

The Scanning Vibrating Electrode Technique SVET370 operates with a non-intrusive scanning, vibrating probe measuring and mapping the electric field generated in a plane above the surface of an electrochemically active sample.

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

Localised corrosion events of less than 5 µA/cm² can be measured with this extremely sensitive technique.

The measurement is performed either on a freely corroding or a dc-biased sample.

SDS370

Voltammetry at the micrometric scale

The Scanning Droplet System SDS370 allows the positioning of a small drop of electrolyte from a PTFE machined 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.

The wetted area is approximately determined by the capillary radius. The small distance between the counter electrode and the sample allows high current densities due to the small ohmic resistance.

The electrolyte drop is then scanned at high resolution across the surface of the sample.



Figure 1: optical micrograph of laser ablated, coil coated, galvanized steel.

SVET370 specifications

SDS head

SDS370 specifications

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. Gain 1-10 ⁵ . Maximum theoretical sensitivity 50 nA FSD. Output time constant 0.1, 1, 10 s
Differential electrometer	10 ¹⁵ Ω 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 0 - 30 µm perpendicular to sample surface
Probe type	Standard SRET™ MkIII platinized platinum probe socket
Electrochemical sensitivity	Better than 5 µA/cm² (using standard PIS test approach)

Note: for the potentiostat specifications, see SECM370, page 4.



SVET image of region shown in figure 1, showing alternating anodic and cathodic behaviour of the defect. The red region is cathodic behaviour, and the blue is anodic



SDS image of polarised partially zinc-coated mild steel sample. Red areas show coating retained intact.

Reference electrode	Ag/AgCl mounted within sensor head
Counter electrode	Pt wire inside the capillary
Micro pump	Peristaltic pump type, 4-channels
Head construction	PTFE head with silicon rubber tubing
Aperture	ID 500 µm, 0.196 mm ²
Resolution	<1 mm depending on solution/surface
Supported techniques	CV, CA, E _{corr} vs. T, LPR, line scan,
	area maps

Note: for the potentiostat specifications, see SECM370, page 4.

SKP370



Surface map of Scanning Kelvin Probe signal recorded over area of sample shown in figure 2.

Surface potential and topography measurements

The Scanning Kelvin Probe SKP370 is a non-contact, non-destructive instrument designed to measure the surface work function difference between conducting, coated, or semi-conducting materials and a sample probe.

The technique operates using a vibrating capacitance probe, and through a swept backing potential, the work function difference is measured between the scanning probe reference tip and sample surface.

The work function can be directly correlated to the surface condition.

Using a capacitance probe also allows to perform topography measurements that can be used as reference for relieving slope or other sample height changes.

0SP370



Non-contact surface profile of electrochemical sensor surface - sample courtesy of Oncoprobe Ltd.

Non-contact topography measurements

Utilising a non-contact laser displacement sensor, the Optical Surface Profiler OSP370 allows fast surface measurement to a very high accuracy.

Features of less than one micrometer can be imaged and measured over a height measurement range of 10 mm without touching the sample surface.

The OSP370 incorporates a CCD displacement sensor mounted on the scanning head of the M370 workstation.

A small spot of laser light at 650 nm is projected down onto the sample surface and the scattered light is focused on to the CCD array allowing the direct displacement measurement of the diffuse scattered light.



Figure 2: optical image of an area of etched zinc coated steel.

SKP370 specifications

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. Gain 1-10 ⁵ . Maximum theoretical sensitivity 0.5 µV FSD. Output time constant 0.1, 1, 10 s
Differential electrometer	10 ¹⁵ Ω 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 0 - 30 µm perpendicular to sample surface
BPC* potential range	±10 V
BPC* DAC resolution	300 μV
BPC* sampling	0.1 to 1000 Hz
BPC* type	PID controller
Probe type	SKPR Tungsten air gap

* BPC: Backing Potential Controller.



OSP head.

OSP370 specifications

Measurement range	10 mm
Reference distance	30 mm
Maximum vertical resolution (static)	100 nm
Spot size	30 µm @ focus
Scan speed	2 mm/s
Multiple readings averaged	Yes
Correct positioning	Red light /green light
Scan range (x, y, z)	70 mm
Light source	650 nm class 2 semiconductor laser max. 0.95 mW



A wide range of accessories developed specifically for scanning probe techniques are available for the M370 system



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.

Cells



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 M370 system.

A range of ultra micro-electrodes (10, 15 or 25 μm diameter) is available for the SECM370 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 M370 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.

M370

- It 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,
- surface maps acquired at up to 70,000 data points in each axis,
- fully programmable macro language for non-standard experiments,
- ASCII exportable data files,
- post data acquisition x, y, z measurement at any point.





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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