Volume 3, Issue 1

Windsor Scientific News

Summer 1998

At Windsor Scientific we have been solution providers to the scientific community since 1989. We have special expertise in Electrochemistry, Surface Measurements and related technologies. We offer the Autolab range of Electrochemical instruments, IBIS Surface Plasmon Resonance and Smart Microsystems Addressable Dot Electrodes.

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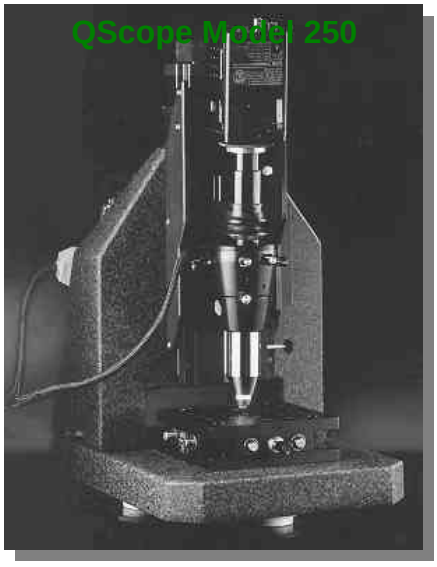


Quesant
VISUALIZE THE NANOWORLD

In London on 7-9th July at the Micro 98 Exhibition, Hammersmith we are launching the Scanning Probe Microscope line manufactured in the USA by Quesant Instrument Corp. Quesant provides a broad range of SPMs with some remarkable breakthroughs in the scanning mechanism, ease of use and value for money.

"Quesant SPMs: versatile, innovative yet affordable"

Whether your work is in surface science, microelectronics, chemistry, life sciences or materials research, there is a QScope in our range at a price that makes sense:-

- **QScope™ Model 250**

A fully featured AFM with an X-Y translation stage accepting samples up to 150 mm square. Includes a video microscope to ease laser alignment and locate sample features.

- **QScope Model 788**

The largest of our SPMs. Designed for semi-automatic operation with a motorised XY stage and 2 video cameras.

- **Resolver**

Capable of handling large samples, this SPM is a lower priced unit. Yet it can be upgraded to the QScope 250 or 788

- **Nomad**

This is a transportable unit that can be placed on any surface or incorporated onto an inverted biological imicroscope.

All are available with WaveMode™, Quesant's unique Intermittent Contact scan mode. Options include liquid scanning and extended Z range. Quesant's ScanAtomic™ software operates under Windows95. The Research Customisation Package provides access to all D/A & A/D signals and enables full customisation of the hardware and access to source code with Visual Basic/C++ software.

WINDSOR SCIENTIFIC LIMITED

AUTOLAB electrochemical instruments.

Windsor Scientific is proud to offer the AUTOLAB electrochemical instruments of Eco Chemie B.V.The basis of these modular instruments consists of a potentiostat/galvanostat, to which optional modules may be added such as a bipotentiostat or an FRA for electrochemical impedance spectroscopy (EIS). This gives you a complete instrument in only ONE box !

The latest release of Windows software for General Purpose Electrochemical Systems (GPES) & Frequency Response Analysis(FRA) has over 40 new features including:-

- Virtual Noise Meters
- Simultaneous display of charge, potential or current
- Multi-channel measurement
- Display total charge
- Save previous scan
- Save scans at regular intervals
- Include shoulders in peak search
- Stop equilibration at threshold current
- Iring vs. Idisk in cyclic voltammetry
- Differential normal pulse in Voltammetric Analysis
- Hydrodynamic Impedance Measurements

New!

µAutolab Type II

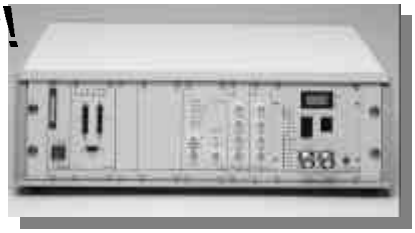
This is a compact, non-modular potentiostat for general electrochemical research and electroanalysis. This single board potentiostat has now been completely redesigned. Brand new features include:

- The incorporation of 16bit configuration throughout (A/D, D/A)
- A new 10nA range giving a current resolution of 30fA
- Galvanostatic as well as potentiostatic modes.
- Supported Techniques such as chronopotentiometry and constant current strippping.

This instrument remains the most cost effective of the range and becomes even more price competitive.

Autolab Electrochemistry Instruments: World-class!

New!



PGSTAT30

This is a new model to satisfy the demands of the new **FRA2 impedance module** for extended ranges up to 1MHz. The bandwidth has now been increased to > 1MHz to accomodate this requirement. All impedance systems delivered from now on with a 30V/1A potentiostat/ galvanostat will include this model. A new range of 10nA is featured giving a current resolution of 30pA.

PGSTAT100

Also joining the PGSTAT series of modular potentiostats is a 100V compliant instrument with a maximum current of 250mA. This is especially suited to measurements in electrolytes with low conductivity (organic solutions).

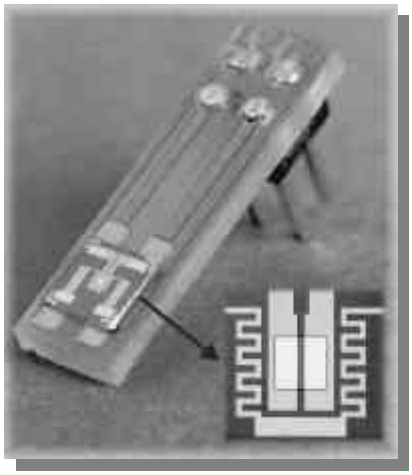
The new models join the **AUTOLAB** range which are all supplied with the GPES software, cables, interface card and manual.

SMART MICROSYSTEMS

SMART microsystems' silicon microdevices give the researcher access to prefabricated, reproducible sensor substrates and microelectrodes for the first time.

Recently, our range has expanded to include devices with coatings that are stable in organic solvents as well as water, giving access to a wide range of non-aqueous chemical systems.

We supply two main categories of addressable electrodes, and have recently extended our range to include heated and interdigitated devices.
Special Offer: a starter pack of electrodes is available!



Microband Addressable Electrodes

Thin film sensors - polymer or solid state gas sensors
Physical Electrochemistry - generation/ detection
Electroanalytical chemistry - array voltammetry
Conductivity Measurement - for solutions or polymer coatings
Polymer Electrochemistry - measurements in polymer films

Microdot Array Electrodes

Sensor Development
Solution Sensors
Physical Electrochemistry - microelectrode studies
Scanning Electrochemical Microscopy

HIGH RESOLUTION SCANNING ELECTROCHEMICAL MICROSCOPY UTILISING A QUESANT AFM SYSTEM.

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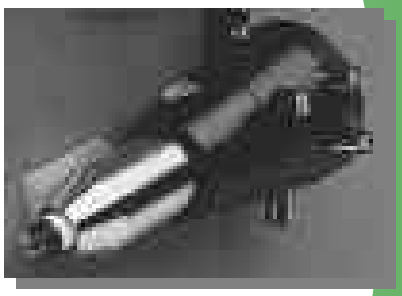
Scanning Electrochemical Microscopy has been used in studies to investigate the local variation of electrochemical reaction currents with a resolution of a few micrometers. This resolution can be further improved if the probe electrode is made smaller and brought closer to the surface. We have adapted a Quesant Resolver AFM to make a SECM of good spatial and temporal resolution.



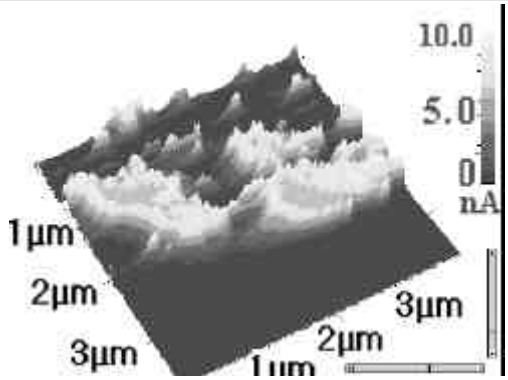
In this commercial instrument, the base is completely open with the scanning mechanism and detection being implemented on the probe mounted above the surface. All of these components are integrated in a 'barrel' shown right exactly like that of an ordinary optical microscope. This 'barrel' can be detached from its granite base support and exchanged for another, without disturbing the specimen. . The sample can be quite large (several centimetres) and can be placed in a normal electrochemical cell (made for example from a beaker) with the surface facing upward. This is extremely convenient for sample preparation and cell arrangement; it enables many applications that cannot use small samples to employ SPM techniques to examine the sample in high resolution.

The solid granite stage construction provides excellent stability during temperature and humidity changes. Quesant's patented optics system allows the laser to track the cantilever/probe, greatly increasing sensitivity and reducing laser light reflections from the sample. It also permits a 90-degree top down view using a CCD camera video system, providing easy laser alignment and positioning of the probe over features that are to be scanned. In addition, lens combinations are available to permit liquid scanning.

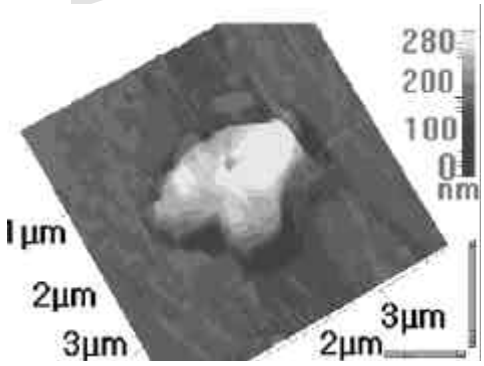
The electrochemical cell was machined out of a PTFE block and the specimen securely mounted horizontally in the base of this block, facing upwards. The SECM 'barrel' then replaced the AFM 'barrel'.

Insulated tips were used as the probe electrode. This enables the probe electrode to be small & sharp. The tip was made from Pt-Ir wire, electrolytically etched in 50%$CaCl_2$ aqueous solution and sharpened in 20% $NaHCO_3$ aqueous solution. The tip was then coated with wax or laquer. This enables the tip to only image the sample area just below it as most of the collected current comes from that area .

SECM utilising a Quesant AFM system



L: High-resolution SECM image of inclusion dissolution from 316F stainless steel:E=+830mV SCE;0.15 mol.dm^{-3} NaCl+0.15 mol.dm^{-3} Na-ClO$_4$.The image was captured 2min after the application of the potential



R: AFM image showing trench developed at the periphery of an inclusion, after the experiment of figure 1, not necessarily in the same location.

The system operates under Microsoft Windows 95 or 3.1. The software is very user friendly and enables the operator to perform even complex operations with ease using menu-driven functions. SECM functions and signal acquisition were implemented into the software using a custom package supplied by the company. This package contains source code of the system software and hardware circuit drawings. The original software was written in Visual Basic and this made the modification of the software by us much easier to suit our specific requirements.
These include:
* Simultaneously recording two current images (one is local current collected by the tip and the other is total surface current collected by the main counter electrode). This enables the comparison between local current and total surface current.
* Precise distance control between tip and sample surface with Angstrom unit accuracy.
* Voltammogram of local area using the tip as a counter electrode.
* Locating the tip to a specific site on any position of the imaged area and marking it electrochemically or mechanically for future referencing purpose, e.g. SEM observation of the same area.

The experimental procedures is as follows: with a sufficient bias on the tip and without solution in the cell, the tip was brought to within tunnelling distance of the surface, to get a reference point for distance control (about 10 Å away from surface). The tip was then withdrawn from the reference point to a precise distance by the piezotube under software control. The solution was then introduced and the SECM imaging carried out at varying constant height. A 2-electrode arrangement was used, with a Pt counter electrode of much larger area than the working electrode, the potential difference between them being imposed by a low-noise ramp generator and the cell current measured by a single amplifier (OPA 111) current-follower. The whole system was placed inside a case that stands on an air table to reduce vibration. This set-up offers a simple but very effective isolation of the system from electrical, acoustic noise and thermal and mechanical vibrations. With the above arrangement, the peak to peak noise level was 0.5 pA and measurements have been performed in the picoamp range. All electrochemical current images were obtained in constant height mode. The images above show some examples.

The authors would like to thank the EPSRC for funding this research.

Ref: "Scanning Electrochemical Microscopic Observation of a Precursor State to Pitting Corrosion of Stainless Steel", Y Zhu and D E Williams,. J. Electrochem. Soc,144, L43 (1997)
"Elucidation Of A Trigger Mechanism For Pitting Corrosion Of Stainless Steels, Using Sub-µm Resolution SECM And Photoelectrochemical Microscopy", David E Williams, Tasneem F Mohiuddin and Ying Yang Zhu, J. Electrochem. Soc, in publica-*