

DC cell analysis techniques Low current DC electrochemical measurements using the ModuLab femto ammeter option

John Harper and Brian Sayers Solartron Analytical, Farnborough, UK. Application Guide: AGML09

Introduction

Measurement of ultra-low current presents a formidable challenge. Measurement technology has not been able to keep pace with the market demand for sensitive, accurate and reproducible low-current measurement instrumentation. Low-current applications include ultra-microelectrode studies, high-impedance coating and corrosion research, nano-electrochemistry and research on carbon nanotubes (CNT). The ModuLab femto ammeter option has been designed to fill this measurement void. This demonstration guide focuses on DC electrochemical measurements. However, the femto ammeter option is also able to measure very high impedance samples (>100 Tohm) when used with a potentiostat and a frequency response analyzer (FRA).

Key system capabilities used in this demonstration

- High resolution signal processing and rejection of noise were fundamental design goals. Measurement
 performance was not compromised with careful selection of components, layout and screening.
- Double screened measurement circuits, triax cabling and high stability reference resistors permit measurements down to the fA region.

Equipment required for this demonstration

· ModuLab potentiostat, femto ammeter option, high impedance demonstration cell and connections

Connections

• For high impedance measurements use 3-terminal connections. Connect the ModuLab potentiostat to the high impedance demonstration box using the connection diagram shown in the following experiment.

Experiment setup

Select "AGML09 Low Current DC Tests" in the "ModuLab Application Guide" project

Step #	Purpose
Step 1	Cyclic Voltammetry using a high impedance resistor e.g. $100~ ext{G}\Omega$
Step 2	Pulse voltage 10 mV using a high impedance resistor e.g. 100 G Ω
Step 3	Pulse voltage 100 mV using a high impedance resistor e.g. 100 G Ω
Step 4	Pulse voltage 1000 mV using a high impedance resistor e.g. 100 G Ω

Additional test possibilities:

- When making extremely sensitive measurements, the DC signal integration facilities provided by ModuLab can very much help to reduce noise. Try experimenting with longer DC integration times in the DC measurement set-up.
- The HV high voltage options can also be used to inject a higher voltage signal into the sample and therefore produce higher current levels. Of course, non-linearity and sample breakdown may occur if the voltage is increased too high, but in some applications this can be a useful technique.
- Impedance measurements. Frequency response analyzers provide a great deal of inherent noise rejection due to the use of signal integration facilities and for low current measurement this is extremely beneficial.

Notes on setup

Consult the setup files in the ModuLab demonstration software for more details.



Data presentation and analysis

Cyclic voltammetry on ultra-microelectrodes is a demanding application. Of critical importance is the ability of the instrument to reject noise which is often several orders of magnitude greater than the signal. A voltammogram of a $100~\text{G}\Omega$ resistor is shown in Figure 1. The sensitivity of the Femto Ammeter card is so great it even has the ability to measure the capacitance associated with the cell cables (as observed in the hysteresis of the I-V plot)!

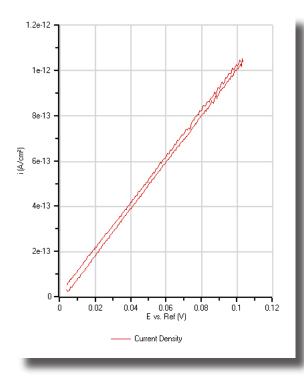
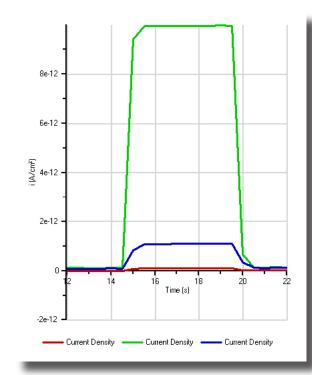


Figure 1. Cyclic voltammogram of a $100 \text{ } G\Omega$ resistor.

Figures 2 and 3 show that even sub-fA pulse techniques produce reliable, noise free results, This will be of particular interest to researchers of nano-electrochemical devices and nano-etching / plating techniques. This will also be of interest to those involved in electrochemical AFM (atomic force microscope) studies.

Figure 2 Applied potential pulse experiments using the femto ammeter demonstration box.

Green =
$$10 \text{ pA}$$
, Blue = 1 pA , Red = 100 fA



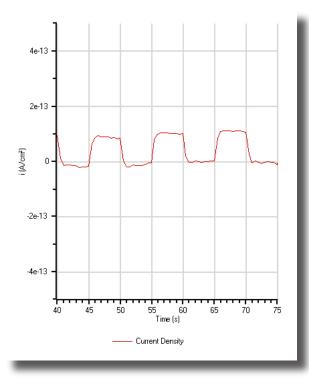


Figure 3. Applied potential pulses of 10 mV across a 100 G Ω resistor showing 100 fA pulses

Conclusions

The ModuLab Femto Ammeter option card represents a major technological step in potentiostat design. Attention to detail, using only the best components and rigorous efforts to eliminate noise have made this option the ideal choice for electrochemical measurements in nanoscale applications and on high-impedance devices.



Solartron Analytical's Quality System is approved to BS EN ISO 9001:1994



...part of AMETEK® Advanced Measurement Technology

UNIT B1 ARMSTRONG MALL SOUTHWOOD BUSINESS PARK FARNBOROUGH, GU14 0NR UNITED KINGDOM

Phone: +44 (0) 1252 556 800 P
Fax: +44 (0) 1252 556 899 F

801 SOUTH ILLINOIS AVENUE

OAK RIDGE TN 37831-2011 USA

Phone: +1 865 425 1360 Fax: +1 865 425 1334

Visit our website for a complete list of our global offices and authorized agents

solartron.info@ametek.com

www.solartronanalytical.com