

Electrochemistry, Electrochemical Engineering and Electrochemical Technology

Southampton, July 5th – 9th 2010

Electrochemistry, Electrochemical Engineering and Electrochemical Technology is a one week, residential course designed to introduce the practical steps essential to implement electrochemical technology on a commercial scale. It is intended to compliment the course *Instrumental Methods in Electrochemistry*, run almost annually in Southampton since 1969. Indeed, we hope that those who found Instrumental Methods in Electrochemistry useful will return to Southampton to attend the new course. The Summerschool also provides formal and informal opportunities for discussion of topics related to the interests of the participants.

In the Southampton tradition, *Electrochemistry, Electrochemical Engineering and Electrochemical Technology* will consist of both lectures (with full written supporting material) and hands-on practical sessions.

The early lectures will cover core material while the final three lecture sessions will address specific topics in electrochemical technology, namely, [PEM Fuel Cells](#), [Electrodeposition for Nanotechnology](#) and [Energy Storage](#). A detailed lecture programme is attached.

All participants will do an introductory voltammetry experiment in the first practical session and then select [two](#) further experiments out of [eight](#) designed to illustrate the core material and the selected specific topics. Details are attached.

For enquiries and registration, please contact: Professor Derek Pletcher, School of Chemistry, The University, Southampton SO17 1BJ, UK. **telephone:** +44 (0)2380 593519 **email:** dp1@soton.ac.uk

Hotels: Participants are responsible for booking their own accommodation. We recommend the [Etap Hotel Southampton](#), the [Hotel Ibis Southampton](#) or the [Novotel Southampton](#). All share a large car park and are located within a few minutes walking distance from the train station, coach station, shopping centres, harbour and city centre. A coach will transport delegates between these hotels and the University

Further details: <http://www.soton.ac.uk/chemistry/seminars/sschools.html>

Programme

Monday July 5th

- 12.00 Registration and Lunch
- 14.00 Understanding Electrode Reactions – Derek Pletcher
- 14.40 Mass Transport – Issues and Solutions – Frank Walsh
- 15.20 Break
- 15.40 Multistep Reactions and Electrocatalysis – Andrea Russell
- 16.20 Introduction to Solid State Electrode Reactions – John Owen

Tuesday July 6th

- 8.30 Bus to University
- 9.00 Introducing Cyclic Voltammetry – Phil Bartlett
- 9.40 Electrode Materials and Membranes – Frank Walsh
- 10.20 Break
- 10.50 Potential and Current Distribution – Derek Pletcher
- 11.30 Electrolysis Cell Design – Frank Walsh
- 12.10 Lunch
- 13.30 Laboratory Session
- 17.00 Bus to Hotels

Wednesday July 7th

- 8.30 Bus to University

PEM Fuel Cells

- 9.00 Introduction to the Science and Engineering of PEM Fuel Cells – Frank Walsh
- 9.40 Characterisation of Electrocatalysts – Andrea Russell
- 10.20 Break
- 10.50 Catalyst Optimisation – Alloy, Particle Size and Support Effects – Brian Hayden
- 11.30 MEA and PEM Fuel Cell performance – Andrea Russell
- 12.10 Lunch
- 13.30 Laboratory Session
- 17.00 Bus to Hotels

Thursday July 8th

- 8.30 Bus to University

Electroplating for Nanotechnology

- 9.00 Fundamentals of Electrodeposition – Derek Pletcher
- 9.40 Applications of Electrodeposition – Phil Bartlett
- 10.20 Break
- 10.50 Nanostructures from Lyotropic Phases – Guy Denuault
- 11.30 Templated Electroplating Using Colloidal Templates – Phil Bartlett
- 12.10 Lunch
- 13.30 Laboratory Session
- 17.00 Bus to Hotels
- 19.00 Dinner at Kuti's

Friday July 9th

Energy Storage

- 9.00 Fundamentals of Lithium Batteries and Supercapacitances – John Owen
- 9.40 Present and Future Lithium Batteries – Matt Roberts
- 10.20 Break
- 10.50 Principles of Redox Flow Batteries for Large Scale Energy Storage – Frank Walsh
- 11.30 Practical (Redox) Flow Batteries for Energy Storage – Richard Wills
- 12.10 Course ends.

Experiment for Tuesday

All participants will do the same experiment in order to ensure that they understand the practice and interpretation of voltammetry. Cyclic voltammetry will be used to study two systems, one involving surface chemistry and the other involving the electroactive species dissolved in the electrolyte.

Experiments for Wednesday/Thursday

Participants will carry out two of the experiments below. They will be asked to select three experiments in order of preference; as far as possible participants will be allocated to their preferred experiments.

1. Charge and Discharge Characteristics of a Flow Battery

Redox flow batteries are a viable technology for energy storage and load levelling in the power generation industry. Normally, a divided cell is used to charge the positive electrolyte in the oxidised direction and the negative electrolyte in the reduced direction. Following connection of a load between the electrodes, the cell can be discharged in a controlled fashion. In the experiment, an undivided flow battery is used, namely the soluble lead acid one. During charge, Pb^{2+} ions are oxidised and reduced to solid PbO_2 and Pb at the positive and negative electrodes. During discharge, the solid phases re-dissolve in the methanesulfonic acid electrolyte. Cyclic voltammetry and constant current charge-discharge cycles will be used to explore the reversibility of the electrode and cell reactions. The performance of the flow battery will be characterised by the voltage-, charge- and energy efficiency. Important variables include current density, electrolyte flow rate and temperature.

2. Characterisation of the Active Area of a PEM Fuel Cell Electrocatalyst

Modern electrocatalysts for proton exchange membrane and direct methanol fuel cells consist of either high surface area metal black materials or supported nanoparticles on a conducting (usually a high surface area carbon). A high active surface area must be coupled with a low catalyst loading (to minimise cost) while achieving a long lifetime and acceptable levels of poisoning by, e.g., CO gas. These challenges are illustrated using 0.2 to 1 mg cm^{-2} Pt on carbon electrode in sulfuric acid. Electrodes will be prepared by painting catalyst inks on to a gas diffusion/base layer. The electrodes will be characterised using cyclic voltammetry to determine the active electrode area (and hence the catalyst utilisation) using (a) hydrogen adsorption and desorption and (b) adsorption and stripping of CO.

3. Characterisation of a Water Electrolyser and a PEM Fuel Cell

The hydrogen economy involved generation, storage and clean combustion of the gas. The water electrolyser and proton exchange membrane (PEM) fuel cell are essential modules. In this experiment, a reversible water electrolyser/ H_2/O_2 fuel cell is used to explore the efficiency of electrochemical energy conversion. The effect of electrode materials and MEAs (membrane electrode assemblies) design will be studied. The cell voltage components of each cell will be measured as a function of current and an intelligent load will be used to simulate changing demands on the cells. Practical issues, such as gas starvation and flow rate control, will be explored.

4. The Electrodeposition of Macroporous Layers

Gold will be electrodeposited into self assembled arrays of microspheres to allow the fabrication of highly ordered microfoams. The influence of a commercial additive on the structure of the deposit and the voltammetry of the plating bath will be investigated. The deposit will be characterised by scanning electron microscopy and voltammetry and the influence of deposit thickness demonstrated.

5. Studies of Mesoporous Electrodeposits

Mesoporous palladium will be electroplated from a plating bath containing a high fraction of a surfactant (ie. a hexagonal phase of a liquid crystal medium). Surface area will be determined using voltammetry in aqueous acid and the properties of the high ordered/high area deposit will be explored.

6. The Technology of Metal Deposition

The deposition of copper is an important technological process in electroplating and electrowinning. This experiment examines the deposition and dissolution characteristics of copper in methanesulphonic acid. A range of complementary techniques is used. The fundamental electrochemistry is studied by cyclic voltammetry at a static electrode and linear sweep voltammetry at a rotating disc electrode. The technology of metal deposition is characterised using a Hull Cell with respect to practical current density range, current distribution and the effect of electrolyte additives on the deposit morphology. The deposition of Cu-Sn (bronze) alloys is explored using a Rotating Cylinder Hull Cell.

7. Investigations of Charge/Discharge Rates of Storage Electrodes

A lithium battery test cell will be constructed using the commercial material LiCoO_2 and piece of Li foil. The effect of cycling rate on available capacity will be investigated using galvanostatic cycling. The influence of the separator and Li interface will be investigated. Diffusional limitations will then be investigated using the Galvanostatic Intermittent Titration Technique (GITT).

8. Electrochemical Impedance Spectroscopy (EIS)

Basic electrical circuits will be constructed and tested using EIS. The recorded patterns will be related to the components within the circuit and real systems. A basic spectrum will then be recorded of a solution redox couple. This will be used to demonstrate how the electrolyte resistance, charge transfer resistances, double layer capacitance and Warburg parameters can be calculated. The impedance of a nanostructured electrode will be recorded and a comparison will be drawn to the transmission line model. The effective resistance of the acid in the pores and the total interfacial capacitance will be calculated. A final experiment will be to analyse the impedance spectrum of a Li ion battery.