

## Introduction to the Foil Cell

### I – INTRODUCTION

The Foil Cell (Part Number: U-uFoilCell), Fig. 1, is an optional accessory for the  $\mu$ TriCell and Shallow  $\mu$ TriCell used with the M370 and M470. It has been designed specifically for use with thin, flat samples, in particular the foil electrodes often used in battery research. Unlike the  $\mu$ TriCell options the Foil Cell allows the user to seal directly onto the sample of interest without the need for any extra components. Furthermore this has been achieved to use a minimal amount of electrolyte, whilst not compromising the user's ability to position the probe with respect to the sample, or perform subsequent experiments.



**Figure 1:** The Foil Cell (U-uFoilCell) allows the measurement of flat, foil type samples.

This application note aims to introduce the Foil Cell through the demonstration of its use with two different sample types, a polycarbonate membrane, and commercial battery electrodes.

### II – ADVANTAGES OF THE FOIL CELL

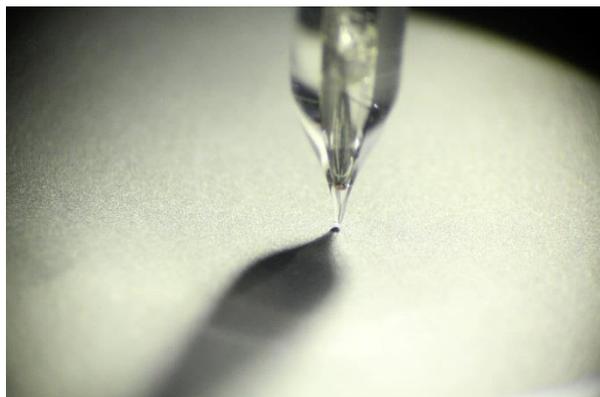
The Foil Cell allows advantages for the user over traditional cell configurations for a number of reasons. When using the Foil Cell it is not necessary to adhere the sample onto the

base of the cell. This is advantageous for samples which are not easily adhered by normal means, for example because they will easily be contaminated by the adhesive, or because the adhesive is not chemically compatible with the electrolyte of interest. Furthermore by removing the need to adhere the sample the setup time for the experiment can be noticeably reduced for the user. By sealing the cell directly onto the sample of interest it also ensures that, unlike in traditional setups, the edges of the sample are not exposed to the electrolyte. This rules out any detrimental effect the edge may have on the measurements performed, and ensures the user is not measuring an edge effect. Finally the reduced electrolyte volume required by the Foil Cell is advantageous when users are working with expensive electrolytes.

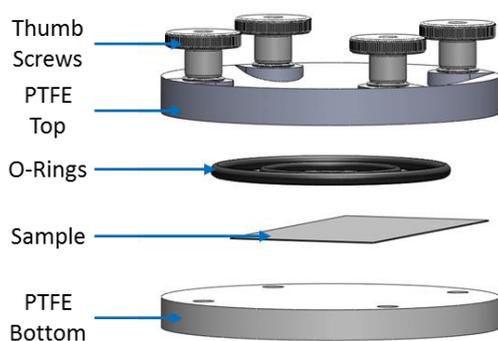
### III – USING THE FOIL CELL

The Foil Cell is an optional accessory which mounts directly to the base plate of the (Shallow)  $\mu$ TriCell. It is composed of a top and bottom PTFE half. During use the sample rests directly on the bottom PTFE half, while the top PTFE half seals to the sample through the use of the sample sealing O-ring. This half has a gradual recess allowing the user to view the probe positioning with respect to the sample using the VCam, Fig. 2.

To use the Foil Cell the user should mount the bottom PTFE half onto the baseplate of the (Shallow)  $\mu$ TriCell, before mounting this onto the M370/470 base. This should then be levelled before placing the sample onto the bottom PTFE half. The top PTFE half should be placed on top, again ensuring the sample is central to the cell opening. Finally all components are held in place by tightening the thumb screws. An exploded view of the complete Foil Cell is shown in Fig. 3.



**Figure 2:** The gradient of the Foil Cell allows the VCam to be angled to position the probe with respect to the sample.



**Figure 3:** An exploded view of the Foil Cell is shown.

While every endeavour has been made to make the Foil Cell relevant to use in battery measurements it is advised that users always ensure the chemical compatibility of all components before proceeding with any measurements.

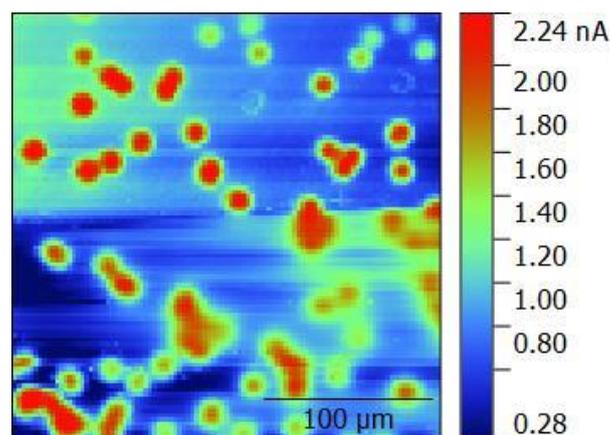
## IV – RESULTS

### IV-1 – Membrane

A Whatman® Cyclopore™ track etched polycarbonate membrane with 12 μm pores (PC12) mounted on top of a piece of copper tape was measured using the Foil Cell and the M470+3300. The copper tape was mounted so a length extended out of the Foil Cell allowing electrical contact to easily be made. SECM area scan measurements were performed in  $2.5 \times 10^{-3}$  mol/L  $K_3[Fe(CN)_6]/K_4[Fe(CN)_6]$  in  $100 \times 10^{-3}$  mol/L KCl using a 10 μm Pt Ultra Micro-Electrode (UME). A Ag/AgCl Reference Electrode (RE) and Pt sheet Counter Electrode

(CE) were used. The probe was biased at 0.65 V vs Ag/AgCl, while the underlying copper tape was biased at -0.25 V vs Ag/AgCl. Measurements were performed over a  $250 \mu\text{m} \times 250 \mu\text{m}$  area with a step size of 2.5 μm, and a scan rate of 20 μm/s. Post processing was performed using the Gwyddion software [1].

The SECM measurement performed on the PC12 over copper tape sample is shown in Fig. 4. A number of dots of 15 – 20 μm diameter can be seen, which relate to the membrane pores. The diameter is larger than the pore size due to the presence of the diffusion field around the exposed conductive area. This allows the probe to sense the conductive region before it is directly over the region.



**Figure 4:** A dc-SECM measurement was performed on a PC12 membrane on a piece of copper tape in  $2.5 \times 10^{-3}$  mol/L  $K_3[Fe(CN)_6]/K_4[Fe(CN)_6]$  in  $100 \times 10^{-3}$  mol/L KCl with a 10 μm Pt UME probe.

### IV-2 – Commercial Battery Electrode Materials

Using the Foil Cell both a commercial positive and negative battery electrode material were measured by ac-SECM. Measurements were performed using the M470+SP-300 system. In both cases area scan measurements were performed in 0.1 mol/L TetraButylAmmonium Iodide (TBA-I) in Propylene Carbonate (PC) using a 10 μm Pt UME probe. A common Pt sheet RE/CE was used. Measurements were performed at OCP with an ac modulation of

100 mV amplitude and a frequency of 250 kHz. At this frequency in 0.1 m/L TBA-I in PC ac-SECM measurements of conductive samples show a reduction in impedance modulus as the tip-substrate distance decreases, allowing for a distinction between conducting and insulating areas. For both samples a 500  $\mu\text{m}$  x 500  $\mu\text{m}$  area with a step size of 5  $\mu\text{m}$ , and a scan rate of 50  $\mu\text{m}/\text{s}$  was used. Post processing was performed using the Gwyddion software.

The resulting area scans performed on the positive and negative electrode materials are shown in Fig. 5 and 6 respectively. In both cases the impedance modulus was lower when measured over the sample surface than when the probe was in bulk, implying a conductive nature. It is likely that for both samples the changes in impedance seen throughout the area scan have arisen from differences in sample topography across the surface. This is particularly the case for the positive electrode material for which localized swelling was apparent on completion of the SECM measurements.

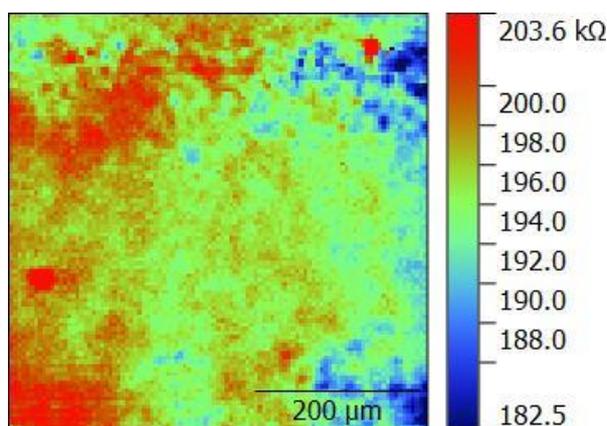


Figure 5: An ac-SECM measurement was performed on a commercial negative electrode material in 0.1 mol/L TBA-I in PC using a 10  $\mu\text{m}$  Pt UME probe.

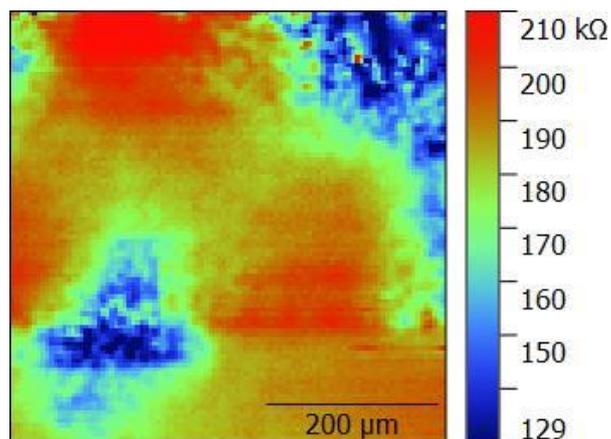


Figure 6: A commercial positive electrode material was measured by ac-SECM using a 10  $\mu\text{m}$  Pt UME probe.

## V – CONCLUSION

The use of the Foil Cell has been demonstrated for two distinct types of flat, foil type samples. Furthermore it was shown that the Foil Cell can be used for both biased and unbiased samples. Finally it was seen that using the Foil Cell instead of traditional cell configurations can reduce the setup time required by the user, whilst giving them confidence the results they measure have not been influenced by edge effects, or poorly insulated samples.

## VI – ACKNOWLEDGEMENTS

The two electrodes measured in this work were purchased by Karlsruhe Institute of Technology (KIT), Institute for Applied Materials - Energy Storage Systems (IAM-ESS). These were provided for measurement as samples of commercial-grade electrode materials.

## REFERENCES

1. [Gwyddion data analysis software](http://www.gwyddion.net)  
gwyddion.net