Simple and Fast Fabrication Methodology of Platinum and Carbon Ultramicroelectrodes (UME) for Scanning Electrochemical Microscopy (SECM)

Emma Mae Lord, Jiju Joseph, Samantha M. Gateman[a,b]*, and Zhifeng Ding[a]*
[a]*Western University, Department of Chemistry, London ON, N6A 5B7, Canada*
[b]*Surface Science Western, 999 Collip Circle, London, Ontario, N6G 0J3, Canada*

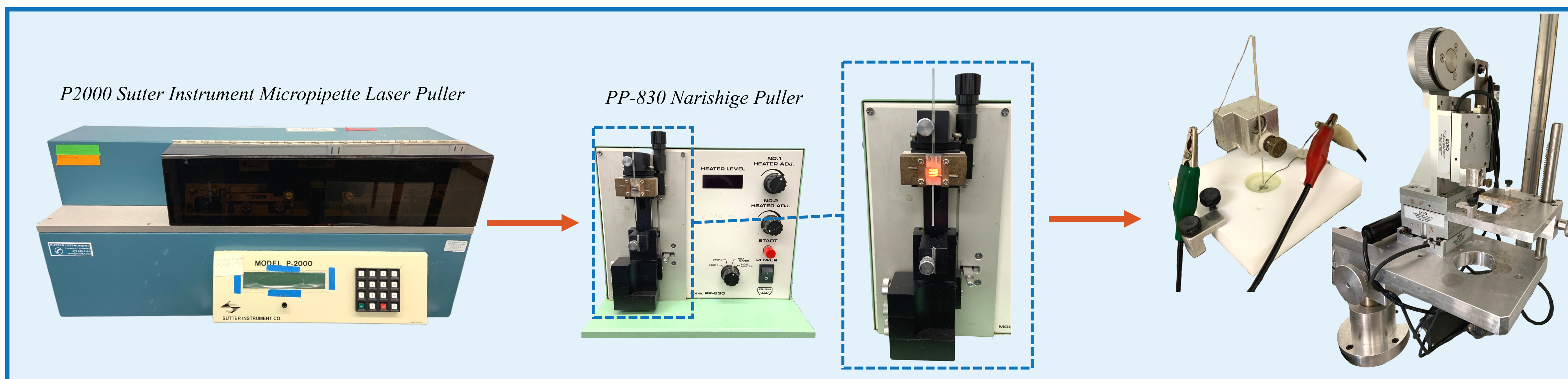


Figure 1. Workflow overview of the instrumentation and setup required for ultramicroelectrode fabrication

Introduction

UME's in SECM are a powerful tool for measuring and imaging microscale features in a system

Problem: Purchasing UME's is expensive and limits customization

Solution: A simple and fast methodology for preparing UMEs was developed

Methodology

1) Attach ~1 cm of electroactive material to copper wire using silver epoxy
2) Insert wire inside Soda-lime glass capillary
3) Transfer into P2000 Sutter Instrument micropipette laser puller
4) Set pulling parameters as follows (adjust heat and pull as required)

340 Heat | 005 Filament | 060 Velocity | 140 Delay | 070 Pull

5) Make connection between electroactive material and copper via silver epoxy
6) Polish using a HEKA micro polisher

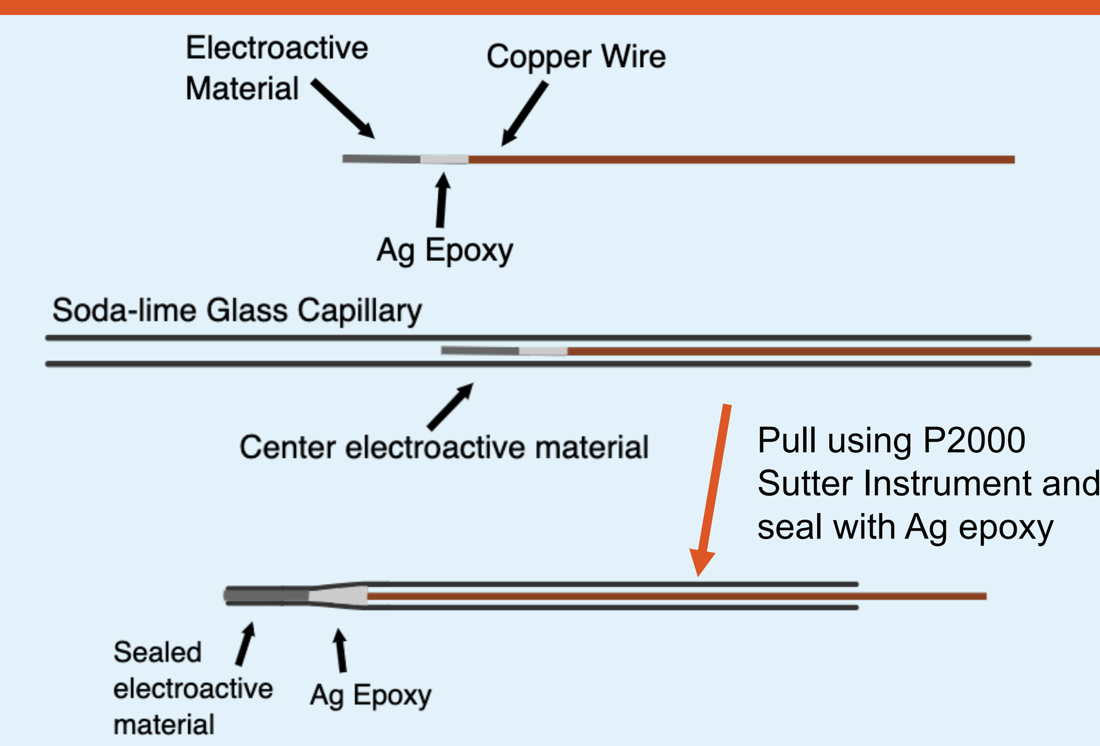


Figure 2. Schematic showing UME preparation

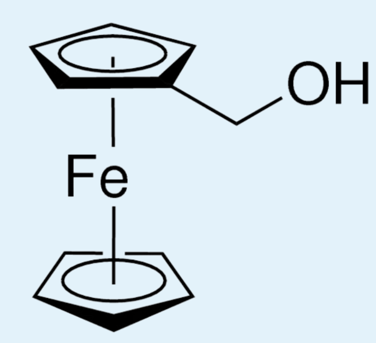
Characterization

Cyclic Voltammetry:

- Used to characterize the electrochemical behaviour of an UME
- Allows for the extraction of the steady state current
- *The more accurate the steady state current, the more stable the UME*[2]

$$i_{ss} = 4nFaDC$$

Steady state current (i_{ss}); Radius of the electroactive surface (a)



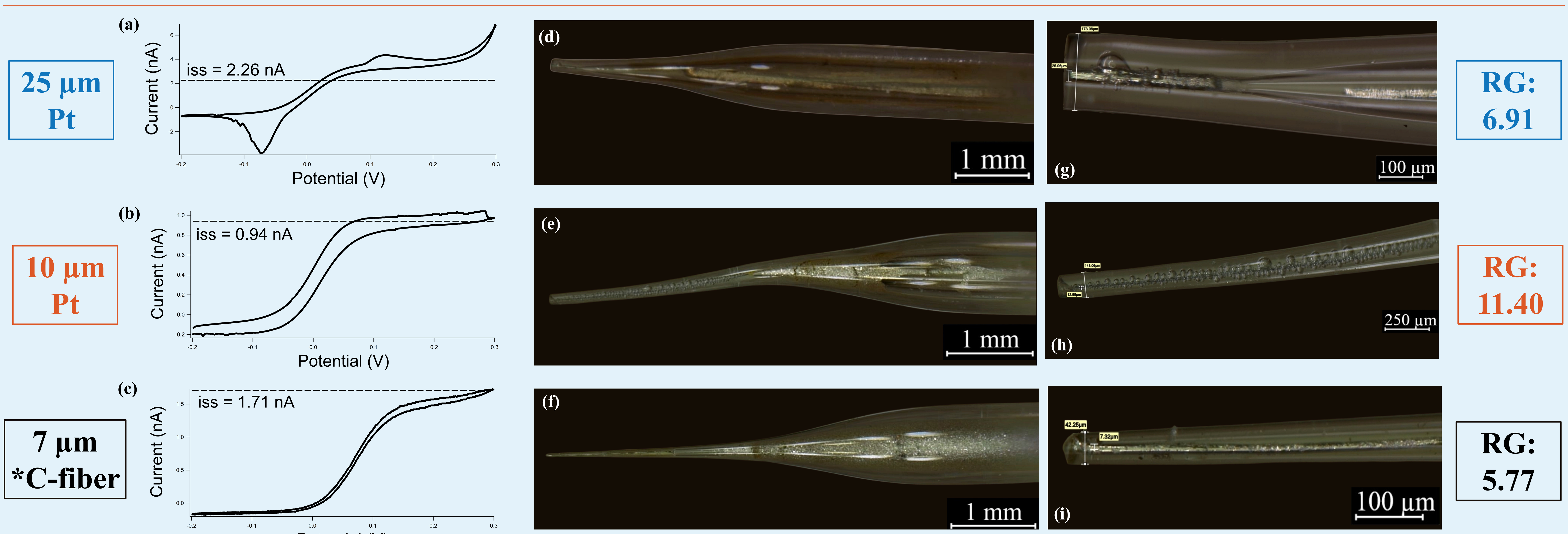
0.9 mM FcMeOH (aq)

Figure 3. Structure of Ferrocenemethanol[1]

Optical Microscopy: (images taken using Leica Optical Microscope)

- Used to determine the RG of the UME
- RG represents the quality of the sealing and polishing of the tip of the electrode
- *The lower the RG value, the higher the quality of the UME*[2]

$$RG = \text{Radius of insulating glass} \div \text{Radius of electroactive surface}$$



*Electrode used for CV made with a different methodology using Borosilicate glass

Figure 2. CVs for (a) 25 µm Pt UME, (b) 10 µm Pt UME, and (c) 7 µm C-fiber. Images (d)-(i) were taken using Leica Optical Microscope of (d) and (g) 25 µm Pt UME, (e) and (h) 10 µm Pt UME, and (f) and (i) 7 µm C-fiber UME.

Applications and Future Work

- SECM is a highly versatile method with many applications such as solar cells, corroding metals, and biological systems
- Future work will focus on improving the quality of the sealing and polishing with the presented method, using fabricated UME's for approach curves and imaging via SECM, and fabricating carbon nanoelectrodes

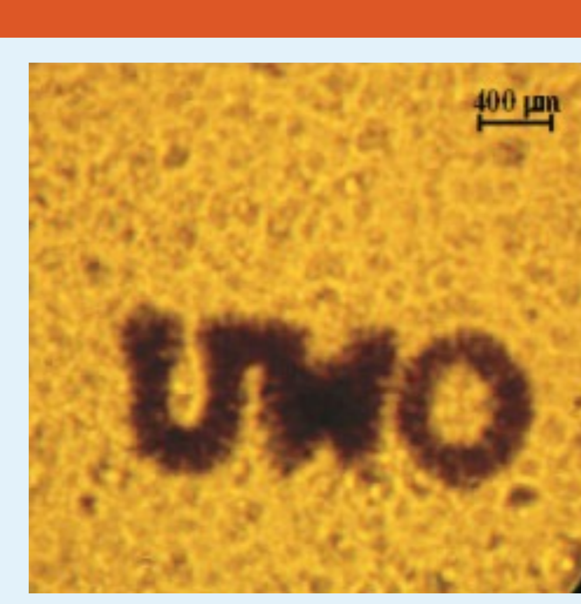


Figure 5. Image of the letters "UWO" taken by SECM[3]

Acknowledgements

I would like to express my gratitude to Dr. Gateman, Dr. Ding, Dr. Joseph, Dr. Filice, Lidia Nemtsov, and all members of RAMPS Western for their guidance and support. As well, thank you to all those involved in the Western USRI Program for making this project possible.

References

1. Ferrocenemethanol Structure, *Millipore Sigma*. https://www.sigmaaldrich.com/CA/en/product/aldrich/335061.
2. Danis, L., Polcari, D., Kwan, A., Gateman, S. M., & Mauzeroll, J. (2015). Fabrication of carbon, gold, platinum, silver, and mercury ultramicroelectrodes with controlled geometry. *Analytical chemistry*, *87*(5), 2565-2569.
3. Zhu, R., & Ding, Z. (2005). Enhancing image quality of scanning electrochemical microscopy by improved probe fabrication and displacement. *Canadian journal of chemistry*, *83*(10), 1779-1791.