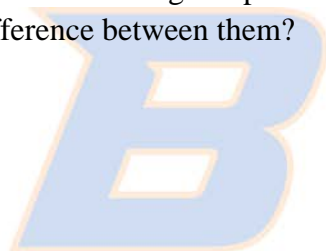


Electrochemical AFM & Scanning Electrochemical Microscopy Exam

General

- 1) It is extremely important to be a good lab citizen when using the EC hardware – i.e., reserving the equipment ahead of time, then rigorously cleaning, rinsing, drying, and returning the sample cell immediately after each use.
 - a. Why are the above measures important?
 - b. Should you ever touch EC sample cell components with your bare hands? Why or why not?
 - c. What is the standard procedure for cleaning the EC sample cell?
 - d. What is the cost of a replacement sample cell?
 - e. What is the replacement cost of the sample chuck, switch block, low noise cable, and spring terminal assembly? When connecting/disconnecting the low noise cable's thin wire leads to/from the spring terminal assembly, where should you grasp the wires and why?

- 2) The cover glass pieces for the electrochemical (EC) sample cell are thin and easily broken.
- What is their replacement cost?
 - When assembling the cell, what can you do to prevent cracking the cover glass? How can you tell if it has a good seal against the O-ring?
 - Are the EC-AFM and SECM cover glass pieces interchangeable? Why or why not? How can you tell the difference between them?



- 3) The EC sample cell comes with three types of O-rings (i.e., different materials).
- What are the black O-rings made of, and what types of samples should they be used for? How much do they cost?
 - What are the white O-rings made of, and what types of samples should they be used for? How much do they cost?
 - What are the red O-rings made of, and what types of samples should they be used for?

- 4) Why and how must you keep track of cover glass and O-ring usage?

- 5) What diameter wire can be used for electrodes? What metals are typically used for the counter (CE) and reference (RE) electrodes, and at what purity level?

- 6) Which electrode (CE or RE) should be bigger/longer?

- 7) The CE and RE wires are inserted through holes punctured by the user in the corresponding sample cell O-ring and pass into the sample cell via a plastic insulating guide. What polymer is this insulating guide/insert composed of? Draw its chemical repeat structure.

- 8) The EC sample cell comes with two inserts made of different materials.
 - a. What is the opaque white insert made of (both its name and the chemical repeat structure of the corresponding polymer)? What types of samples should it be used for?

 - b. What is the translucent/transparent white/clear insert made of? Give both its name and the chemical repeat structure of the corresponding polymer. What types of samples should it be used for?

- 9) What are the sample size requirements/restrictions for EC-AFM and SECM?

10) Briefly describe how to prepare an Ag/AgCl RE using silver wire and confirm the process was successful.

11) What do you need to provide in terms of syringes, micropipettes and tips, glassware, and electrolyte solution (both quantity and filtration)? How much fluid does the sample cell hold? What can happen if you add too much?

12) Can aqueous electrolyte solutions be used inside the glovebox?

13) Where do the four BNC connectors on the opposite end of the “Signals” cable from the bipotentiostat attach to the NanoScope V Controller?

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14) When (i.e., for what experiment types) is the switch block attached to the sample stage switched to the left? To the right? Explain why.

15) What temperature range is accessible with the tungsten alloy heater integrated into the SECM chuck and the Lakeshore Model 335 Temperature Controller connected to the MUX box?

16) Do the Nanoscope and CHI software programs communicate directly with each other (i.e., know what the other is doing/what settings have been selected)?

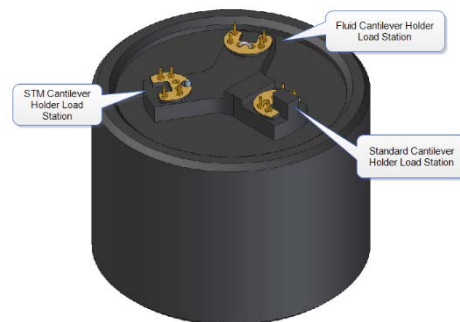
- 17) Why is it important to verify and update the Blind Engage position every time you perform an EC-AFM or SECM experiment (including whenever you change probes and/or remove the sample cell)? What are typical XY values for EC-AFM and SECM, respectively?

EC-AFM

- 18) What AFM probe type(s) are typically used for EC-AFM, and what is the resultant lateral spatial resolution? What is the approximate cost of these probes?

- 19) Why are there 2 different EC-AFM probe holders (i.e., what are their uses)? Are they interchangeable? What are their respective prices?

- 20) A picture of the probe holder mounting block is shown below. Which of the 3 probe loading stations should be used with the EC-AFM probe holder and why? Why is it important to check to ensure all 4 metal pins are present and straight (i.e., not bent) prior to *gently* placing the probe holder on the appropriate loading station (i.e., not forcing it onto the pins)?



21) Connecting the various electrode wires can be confusing, but is crucial to properly setting up the experiment.

a. Give the colors and identities (i.e., connectivity) of the 4 posts on the spring block terminal.

b. How is biasing of the sample accomplished (i.e., what wire is connected where)?

22) When is the MUX box used in EC-AFM?



23) What is the size and shape of the available sample scanning area in EC-AFM?

SECM

24) The SECM nanoelectrode probe is the heart of PF-SECM.

a. What is the approximate diameter of the SECM probe?

b. What is the resultant lateral resolution, both for topography and electrochemistry?

c. How much do SECM probes cost?

25) Mounting the SECM probe on the special PF-SECM probe holder is tricky.

- a. Can you load an SECM probe on the SECM probe holder using the normal (black) probe mounting block?
- b. Which tweezers must you use to load the SECM probe on the SECM probe holder?
- c. What precautions must you take to minimize the chances of destroying the probe via ESD?

26) Connecting the various electrode wires can be confusing, but is crucial to properly setting up the experiment, and is somewhat more intuitive for SECM than EC-AFM.

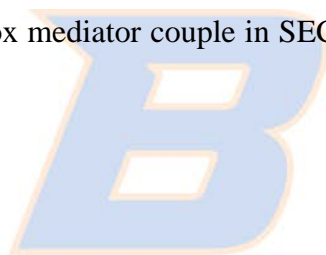
- a. What do working electrode 1 (WE1) and working electrode 2 (WE2) correspond to in the context of SECM?
- b. Give the colors of the 4 posts on the spring block terminal and identify their connectivity (i.e., name the corresponding wires/leads).
- c. Why are there two blue wires? What are they used for?
- d. What is the yellow wire coming from the special low-noise SECM cable for (i.e., what is its name) and where does it go (i.e., where is it connected/plugged in)?

27) Why must you make all electrical connections, power up the bipotentiostat, and start up both the Nanoscope and CHI control software prior to plugging the SECM probe cord into the bottom of the strain release module?

28) By how much and in what direction is the available sample scanning area offset from center in SECM?

29) What is the size and shape of the available sample scanning area in SECM?

30) What is the purpose of the redox mediator couple in SECM? How do you go about selecting an appropriate pair?



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31) When is the MUX box used in SECM?

32) What sorts of current levels are expected in SECM for WE1 (i.e., the SECM probe)?

33) How do you set up biasing of WE1 (i.e., the SECM probe) in the CHI software and measure the resultant electrochemical current? What must you *always* remember to do before withdrawing the probe from solution (and why)?

34) How do you clean the SECM probe after use?



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