Chem 524 - Experiment 0
Making and cleaning a Pt working electrode

Making your Pt microdisk electrode:

Obtain a ~1 cm long piece of Pt wire and a longer piece (10 cm) of bare copper wire. Both are available in our laboratory. The diameter of the Pt wire will determine the area of your finished electrode. Pt wire in the range of 0.25 - 1.0 mm works well. We typically use 26-28 gauge copper wire.

Solder the end of Cu wire to the end of the Pt wire using rosin-core solder. Be careful not to get any solder near the other end (the working end) of the Pt wire.

Encapsulate your soldered wire in a section of 3-4 mm OD glass tubing as shown, so that the Pt is protruding slightly from one end and the Cu wire from the other.

Mix a fresh batch of Loctite 1C Hysol Epoxi-Patch adhesive on a disposable surface, such as a plastic weighing boat. To mix the epoxy properly, you should squeeze out equal ~ 2 cm lengths (not equal amounts) of parts A and B. This particular brand of epoxy is electrochemically inactive and quite resistant to organic solvents when cured. Cover the Pt wire completely with the epoxy, and also push some epoxy into the open end of the glass tube so that it covers the solder joint.

Let the epoxy harden at room temperature for several hours. When it is no longer tacky, cure it in the drying oven (80-110 °C) for one hour.

Sand the end of your electrode with sandpaper to expose the Pt disk. You may sand all the way down to the glass, but it is not necessary. Finish the job with polishing cloth and alumina polishing grit, ending with the finest polishing grit available (0.05 µm).

You should examine the end of your masterpiece in the optical microscope to confirm that the Pt disk is exposed and covered on all sides with epoxy.

Cleaning your Pt electrode:

Your polished electrode looks clean, but it is still covered with both organic and inorganic "goop" from the polishing process.

Sonicate the Pt end in deionized (DI) water and then rinse with DI water and allow to air dry.

Next we will clean the electrode surface electrochemically in a solution of 0.5 M aqueous H₂SO₄.
Obtain a saturated calomel reference electrode (SCE) and Pt counterelectrode from your instructor. Rinse them with DI water and put them into a one-compartment cell (a small beaker or test tube works well) with your working electrode. Connect the three leads from the potentiostat to the three electrodes.

The first step in cleaning the working electrode is to hold it at +2.0 V vs. SCE for 2 min. It should evolve oxygen, and hydrogen will evolve from the counter electrode. This process burns off any organic residue and also anodically dissolves trace metals (such as Pb and Sn from your solder).

Next, cycle the electrode between -0.23 and +1.10 V vs. SCE at 100 mV/s, stopping after 10-20 cycles at the positive limit (+1.10 V). At the end of the process, you should get a CV that looks like the one below, shifted slightly on the potential axis because you are using SCE rather than RHE as the reference:

The anodic waves near the positive limit correspond to oxidation of the Pt surface, and the large cathodic wave at +0.7 V (vs RHE) corresponds to stripping the oxide to regenerate the elemental Pt surface. Initially, this wave will be very large, because you grew a thick oxide at +2.0V, but upon repetitive scanning the CV will start to look like the one above. The cathodic and anodic waves near the negative limit of the scan correspond to adsorbing H atoms onto the Pt surface and stripping them off. A clean Pt surface will show these features and none other. If you have extra peaks in your CV, discard the H₂SO₄ solution and start over with the anodization step in fresh 0.5 M H₂SO₄. Once you have a CV that looks good, stop the scan at 1.10V, remove your electrode from the solution, rinse it with DI water, and allow it to air dry. You now have a clean
Pt electrode with a monolayer of oxide on the surface, which is ready for cyclic voltammetry or other experiments.

You can determine the geometrical area of your electrode from the diameter of your Pt wire \( A = \pi r^2 = \pi d^2/4 \). Compare this number to the value you get from the double-layer capacitance, which can be estimated from the non-Faradaic current in the "double layer" region at +0.45 V vs. RHE in the figure above. Here the difference between the anodic and cathodic currents is \( 2vC_{dl} \), where \( v \) is the scan rate in volts/second and \( C_{dl} \) is the double layer capacitance. For clean Pt at this potential in \( \text{H}_2\text{SO}_4 \), the capacitance is on the order of 50 \( \mu \text{F/cm}^2 \). You can also obtain an estimate of the surface area by integrating the charge under the hydride adsorption-desorption waves. The ratio of the electrochemical to geometrical area is a measure of the roughness of the Pt electrode.