

Protocol for Electropolishing (See Ref. in Tao's Thesis)

- 1) Use Au or Pt wire as both the reference and counter electrodes.
- 2) Use 1-3 M H₂SO₄ as the electrolyte.
- 3) Increase the potential to a high value (~2 V). The Au surface will start to bubble. Sit at this potential for a couple of minutes. The surface will turn red due to the formation of Au oxide.
- 4) Wash with H₂O and put a drop of conc. HCl on the surface and the red oxide will be removed.
- 5) Clean the surface in piranha.
- 6) Electropolishing only needed for the removal of noble metals (Pt, Pd).

(You could also consider sputter cleaning the crystal in the UHV prep chamber. This should only be done as "last ditch effort" if you are worried that the crystal is significantly damaged.)

Other Important Tips

- Control the potential of the surface (Turn on the potentiostat and set desired value) before plugging in the scanner.
- Plug in the scanner before dipping the tip into solution. Before tunneling, change the tip potential for both W and Pt/Ir tips to reduce the leakage current. W tips should be kept at a value near -0.6 V and Pt/Ir tips should be near 0.05 V.
- Try scanning without the o-ring to reduce noise.
- Only need to flame anneal the single crystal for 2 mins. The color of the crystal should be a reddish-orange color.
- Perchloric acid is a much better electrolyte than sodium perchlorate. You have no control over pH in NaClO₄.
- Pt electrode is more stable in HClO₄.
- The difference between Pt and Ag/AgCl in HClO₄ is ~0.55 V (We measured it to be 0.47 V).
- The difference between SHE and Ag/AgCl is ~0.2 V.
- Tip bias is the potential difference between the tip and the working electrode.
- The tip potential is the difference between the tip and the reference electrode.
- $E_{\text{tip}} - E_{\text{sample}} = \text{Tip Bias}$
- Use the old single crystal to measure the correct distance for the tip to save time in tip approaches when the fluid cell is used. Use a ruler to measure the length of the tip so you can make all subsequent tips the same length.
- Can slow down the tip approach speed to avoid tip crashes.
- The noise level for Pt/Ir tips is ~20 pA. For W tips, it is closer to 10 pA.
- Use quite small "stop at" values (0.3 nA) for the tip approach to avoid crashes.
- To help reduce drift, stop scanning for awhile after the tip approaches. Also, try letting the sample plate sit in quiet room to thermally equilibrate before approaching.

- If not all of the wax is off of the wire that goes into the scanner, you could see some drift due to the compression and expansion of the wax. Also try cutting the extra wire from the MI Pt/Ir tips before scanning to reduce drift.