

Protocol for cleaning glassware, Teflon bottles, electrodes (Pt and Pt/Ir), fluid cell (Teflon part), and single crystal

- 1) Anything that comes into contact with the single crystal, Pt and Pt/Ir electrodes, or fluid cell **MUST** be cleaned with the following procedure. It is a good idea to keep the single crystal in a separate beaker from the other components to avoid scratching.
- 2) Push the Teflon part of the fluid cell out of the base for cleaning. Never clean the base in piranha b/c Al will leak out and cause the base to be corroded.
- 3) Begin with a treatment in piranha (1:3 H₂O₂ (30%): H₂SO₄). **WEAR GLOVES!!** (If crystal has come into contact with Ag, it must **first** be cleaned with concentrated HNO₃ to remove the Ag.) Try to use an amount which covers almost all of the exposed surfaces. It is fine to leave in piranha overnight, just be sure that the generated gas can be vented.
- 4) Remove the piranha and rinse 8X with nanopure H₂O **DIRECTLY** from the source (go to the P-Chem lab). The piranha should be put down the drain with lots of water. It is **NOT** safe to store piranha.
- 5) Sonicate in beaker for ~5 min. in acetone/MeOH/EtOH (Any one of these solvents is acceptable.)
- 6) Rinse 8X in nanopure H₂O **DIRECTLY** from the source.
- 7) **NEVER** touch the top of the single crystal with the tweezers.
- 8) If using the metal tweezers, make sure they have been cleaned in acetone and passed through a H-flame. The metal tweezers **CAN NOT** be cleaned in piranha.
- 9) The ceramic tweezers can be cleaned in piranha just make sure they are blown dry in the direction away from the metal so that it does not get corroded.
- 10) All electrolyte solutions **MUST** be made from the most pure neat chemicals available and must be made in Teflon bottles to avoid contamination. (OPTIMA brand from Fisher or JT Baker)
- 11) Clean the Ag reference electrode by sonicating in acetone and nanopure water.

Protocol for taking CV's

- 1) Begin purging the environmental chamber with N₂ (g) ~1 hour before taking CV's.
- 2) Place a Petri dish with nanopure H₂O in the bottom of the chamber so that the electrolyte does not evaporate.
- 3) Monitor the flow of N₂ (g) to ensure that it is flowing at a steady rate. Be sure to check it after the first 5 mins.
- 4) Be conscious of the type of electrolyte used and the corresponding quasi-reference electrode. For acids, use Pt electrode. For basic/neutral, use a sealed Ag electrode.
- 5) Use CV's as a diagnostic tool to see the condition of the clean Au surface. (See Hamelin paper (A. Hamelin. J. Electroanal. Chem., 407 (1996) 1-11.)) A nice CV is saved as 2-1-08AuCV8 in AuCV folder. Can see reconstruction peaks. May want to sweep the potential multiple times to fully clean the surface of contaminants.

- 6) If the clean surface CV's do not look good, try oxidizing the Au surface to remove impurities. Go to 0.7 V or higher then sweep to 0 V and back at 0.05 V/sec. Then pulse down to 0.2 V for 2 sec. Then go to 0 V.
- 7) When taking CV's with Pt as reference, don't go beyond -1 V b/c you can get close to hydrogen peak which will change the Pt electrode.
- 8) When first beginning to take a CV on a UPD system, look for the bulk deposition peak to get a gage on what potentials you want to scan, although don't add solution at a potential that will immediately cause bulk deposition.
- 9) When you use faster sweep rates, you will see larger current peaks.
- 10) Should NEVER see bubbles around the reference electrode because the reference electrode should not be reacting.
- 11) Prior to starting a CV, turn the controls off and on in the software. If still having problems getting good CV's, try restarting the computer, control box, and software. There is definitely a problem with the MI software.
- 12) The bulk deposition peak will not be symmetric if other facets other than the (111) are present, which is the case for our single crystal. (Use of an o-ring may help.)
- 13) Change gains on the software AND potentiostat if peaks are too large or small.
- 14) Some programs that can be used to integrate the peaks in the CV are MatLab, Origin, or Egor.
- 15) After running quite a few CV's, the surface has many vacancies and islands and is not very flat due to the nature of taking a CV.
- 16) Remove Petri dish of water before trying to scan as humidity may affect scanning. Also, stop the N₂ purging.

Details on piranha solution

Piranha solution may be prepared by **adding the peroxide to the acid**. The hot (often bubbling) solution will clean organic compounds off substrates, and oxidize/hydroxylate most metal surfaces. Cleaning usually requires about 10 to 40 minutes, after which time the substrates can be removed from the solution. Due to the self-decomposition of hydrogen peroxide, piranha solution should be used freshly-prepared. Piranha solution should not be stored. Piranha solution can be explosive. Mixing the solution is exothermic. The resultant heat can bring solution temperatures up to 120°C. Explosions may occur if the peroxide solution concentration is more than 50%. A 30% peroxide in water solution is more reasonable.

Clean Au(111) CV (File: 2-1-08AuCV8)

