Standard Operating Procedure: Electrode Polishing and Care

Last updated in 05/15/17 by Selene Li

A smooth, clean, and uniform electrode surface is necessary for reproducible problem free electrochemistry experiments.

Examine The Electrodes:

- An electrode with deep scratches may need to be wet-polished on the 800 grit sand pad.
 - Put a few drop of water on top of the sand pad, and polish the electrode in a figure 8 motion, keeping the electrode facing parallel to the polishing pad for 5-10 minutes. After that, rinse off the electrode with DI water.
 - This polish procedure is very aggressive, please see appointed person for questions first before proceeding.
- An electrode with visible scratches or dirt should be polished starting from Aggressive Cleaning.
- An electrode, which has a perfectly smooth mirror shine surface, should still be polished prior to use, starting with Routine Cleaning.
- In many occasions, electrode polishing procedure is strongly based on judgments off previous experience. If questions arise, please see appointed person.

Reagents and Equipment:

- Squirt bottles for water, MeOH, and isopropanol
 - The diamond polishing slurries $(15-\mu m \text{ blue}, 3-\mu m \text{ yellow}, \text{ and } 1-\mu m \text{ white})$ are oil base, so a MeOH flush of the electrode is required.
- 2-50mL beakers for sonicating electrodes, which are in the E-Chem draw with the polishing kit.
- 1 larger beaker for waste
- Polishing kit 2 boxes (in the E-Chem drawer), containing four polishing pads, three white diamond (nylon) pads (15 μm, 3 μm, and 1 μm), and one fuzzy brown alumina pad, and both diamond (15 μm, 3 μm, and 1 μm)and alumina polishing slurry bottles.
 - Shake polishing slurries thoroughly before use.

Aggressive Cleaning:

- 1. Rinse the 15-µm nylon polishing pad thoroughly with DI water prior to use. This will help break up any clumps of grit that were not rinsed off from a previous use.
- 2. Rinse the electrode surface with DI water followed by MeOH to flush away any encrusted material on the surface.
- 3. Start polishing with the largest particle size (the blue 15-µm diamond polish)

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- Find the 15-µm polishing pad. It will be labeled on the back, and have a blue spot on the front, if it has been used.
- Shake the bottle of 15-μm diamond polish thoroughly and apply threefour drops in the center of the pad.
- Polish the electrode in a figure 8 motion, keeping the electrode facing parallel to the polishing pad for 5 minutes.
- After that, rinse the electrode well with MeOH.
- Check the electrode face for any scratches.
 - If new scratches appear, this means there were larger particles on the polishing pad.
 - In this case, re-rinse the polishing pad and repeat the polishing steps.
 - If scratches still appear, you may consider replacing the polishing pad with a new one and start the polishing procedure over from the largest particle size. Recommend to see the pointed person before replacing new pads.
- Sonicate the electrode for 5 minutes in a beaker clean with fresh and clean isopropanol to remove any residual particles.
- Sonicate the electrode for 5 minutes in a beaker of fresh and clean DI water.
- You may have to repeat this process a few times until no new scratch.
- 4. Repeat the polishing procedure described in Step 3 above with the yellow 3 μm diamond polish with appropriately labeled polishing pad.
- 5. Repeat the polishing procedure described in Step 3 above with the white 1 μ m diamond polish with appropriately labeled polishing pad.
- 6. The final polishing step is with the alumina polish
 - Rinse the fuzzy brown pad thoroughly with DI water.
 - Shake the bottle of alumina polish well and put four-five drops evenly around the pad surface.
 - Polish the electrode in a figure 8 motion, keeping the electrode facing parallel to the polishing pad for 5 minutes.
 - After that, rinse the electrode well first with DI water.
 - Sonicate the electrode for 5 minutes in a beaker of clean water to remove any residual particles, and then rinse the electrode again with DI water.
 - Check the electrode face for any scratches.
 - If new scratches appear, this means there were larger sized particles on the polishing pad.
 - If this is the case, replace the polishing pad with a new one and start the polishing procedure over from the largest particle size.
 - If the scratches stay, go back to use the white 1- μ m diamond polish and the alumina polish.
 - Rinse the electrodes with MeOH afterward.

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Routine Cleaning:

If an electrode has a perfectly smooth mirror shine with very light scratches on the surface, it should be polished starting from $3-\mu m$ yellow, $1-\mu m$ white diamond polish, and finally the alumina polish with appropriately labeled polishing pads. Repeat Step 1-6 above for detail procedure.

Before using the electrode:

- Soak in clean and fresh isopropanol for an additional 10-15 minutes
- Soak in the solvent, which will be used in echem data collection, for 10-15 minutes
- Allow electrodes to dry at room temperature or by blowing N₂ from a distance, and wrap the electrodes using lens papers to protect the electrode surface. NEVER use a kimwipe.
- **DO NOT** touch the electrode surface with your fingers, or place the surface in contact with sharp objects or other materials which could scratch it.

After using the electrode:

- Rinse the electrode at least 3 times with the clean and fresh solvent, which was used for the echem measurement.
- Allow the electrode to dry at room temperature or by blowing N2 from a distance. Place the electrodes back the containers, where the electrodes are originally stored in.

If you have any questions, see appointed person.

Personal Protective Equipment:

Safety glasses, nitrile gloves, and lab coats designated for electrode polishing are required.

Waste Disposal:

Any hazardous waste, including diamond and alumina polishing paste, should be disposed properly according to the standard operating procedure.

Hazards:

Solvents, polishing reagents and glassware can be dangerous to you. Please handle them with care.

Material Safety Data Sheets:

Call the ORCBS (517-355-0153) or see lab copy for samples in questions if available.