

## Protocol for Tip Etching and Wax Coating of W Tips

- Make a loop of Pt/Ir wire (diameter of 0.25 mm). Push loop into a 22 gage hypodermic needle, which is first pushed through a rubber stopper. The rubber stopper should be held in a clamp. Pt/Ir loop must be round and flat.
- Cut off the angled end of the needles.
- Use a needle stuck into another rubber stopper to catch the other part of the wire, so that two tips can be obtained rather than just one.
- Push the W wire through a 22 gage needle to keep it straight. The wire should be as perpendicular as possible to the meniscus in the loop. The needle and wire are also in a rubber stopper which is connected to a clamp.
- Pour the freshly-made 3 M KOH into a vial to use to form the lamellae on the loop. Dip twice in the beginning. If the meniscus breaks near the start of the process, gently clean loop with a KimWipe.
- Connect loop and W wire to the variac. Use a voltage between 3-5 V on the variac. Change the voltage according to the amount of bubbles you see in the loop. You want to see a fair amount but do not want large bubbles that break the film. It can take up to 20 mins. for a tip to etch.
- When the W wire starts to get thin, turn the voltage down slightly.
- As the neck thins, use a thinner film on the loop if the lamella breaks. (Don't dip more than once.) When dipping make sure to not change the position of the loop with respect to the W wire. The position of the meniscus should be kept constant.
- As the wire gets very thin (Look at wire with an optical loop), keep an eye on the wire. As soon as the bottom of the wire drops, turn off the variac. If this is not done immediately, the top tip will continue to be etched and not be as sharp and should not be used.
- Once the tips are etched, wash them (Etched end should always be pointed up.) in nanopure H<sub>2</sub>O and ethanol before coating them with wax.
- The etched tips should be waxed within the same day. Heat the paraffin wax to 70°C in the oven. The wax should look clear and the wax needs to reach to the top of the beaker so that you don't have to dip too far into the beaker. It may be helpful to stabilize your wrist for the dipping procedure. If the tip is dipped immediately after taking the wax out of the oven, you may need to dip twice quickly. If some time has passed and the wax has begun to cool, only dip once. Bubbles of wax on the tip are not good.
- If too much wax gets onto the tip, you can remove it with the hot air gun and start over.
- Keep wax covered when not using.
- To check the stability of the faradaic current on the waxed tips, look at the oscillations in the tunnel signal monitor. Don't want to see large oscillations. Can change the values of the axes to make more sensitive. Keep the tip potential around -0.6 V for W tips and ~0.05 V for Pt/Ir tips. See how the leakage current responds to changes in the tip potential. Good tips will respond immediately meaning that the very end of the tip is metallic, but the overall leakage current should remain small (<70 pA.).

- W tips are good for imaging molecules and may help to reduce the noise level even further from what is seen with Pt/Ir tips.

Tip Etching Set-up



