

# Tungsten Tip Preparation

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## General aspects and observations:

When performing STM measurements, the stability of the tip plays a crucial role. Therefore, a sharp tip which comes to a point rapidly is desired (see Fig. 1). With the aim of achieving these characteristics, several concentrations and applied voltages have been tried. By experiment and SEM image acquisition, it has been found that the optimal parameters for etching are a solution concentration of 8g NaOH/100ml distilled water and an applied voltage of roughly 4V.

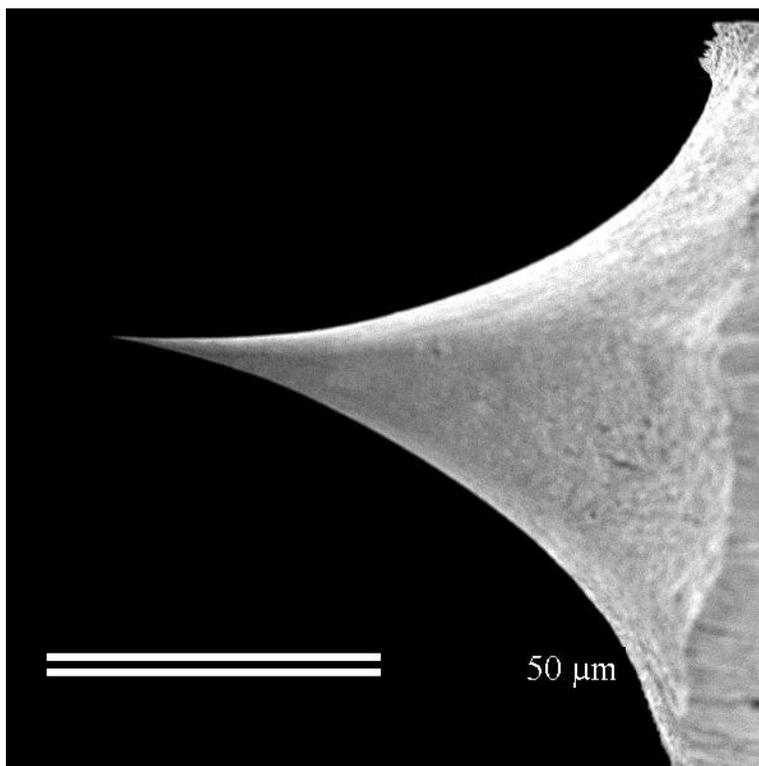


Fig. 1: SEM image of a Tungsten tip prepared in 2M NaOH solution (8g/100ml) with an applied voltage of 4.2V

## Equipment:

- *tip holder* (see Fig. 2);
- *variable voltage power supply*;
- *etching station* (see Fig. 3);
- *electronic scale*;
- *optical microscope* - offers a quick view of the quality of the tip (also it is a good way to check if the tip touched something after the etching and cleaning processes were completed);
- *ultrasonicator* (*ultrasonic cleaner*);
- *beakers*;
- *glass stirring rod*;
- *tweezers* – ‘curve tips’ and ‘very fine rounded points’ types of tweezers have been used (see Fig. 5); ordered from Techni-Tool;
- *pliers* - useful when: i) *bending the tip*, in which case the best to use are the short chain nose head pliers and ii) *inserting the tip into the holder*, in which needle nose or long chain nose are the best choice; both types of pliers have been ordered from Techni-Tool;
- *cutters* - one thing worth mentioning here is that the choice of the cutters is important because of the following: when one cuts the wire from the roll, it can split at the end, making it more difficult to bend the wire and mount it onto the holder; this can be avoided by choosing cutters with full flush cutting edge; ordered from Techni-Tool;
- *multimeter* – for measuring the AC voltage which was applied between the electrodes.

## Materials:

- *Tungsten wires* – ordered from ESPI METALS; 0.02’’ in diameter;
- *NaOH 2M grains* – ordered from ALFA AESAR;
- *distilled water* – obtained from OU Chemistry Department;
- *Isopropanol*;
- *flexible tubing (made of thermoplastic material; useful because it is not affected by the etching process)* – ordered from COLE-PARMER Instrument company; dimensions: 0.020’’ ID, 0.083’’ OD, 0.0315’’ WALL;
- *graphite electrode* – obtained from the machine shop.

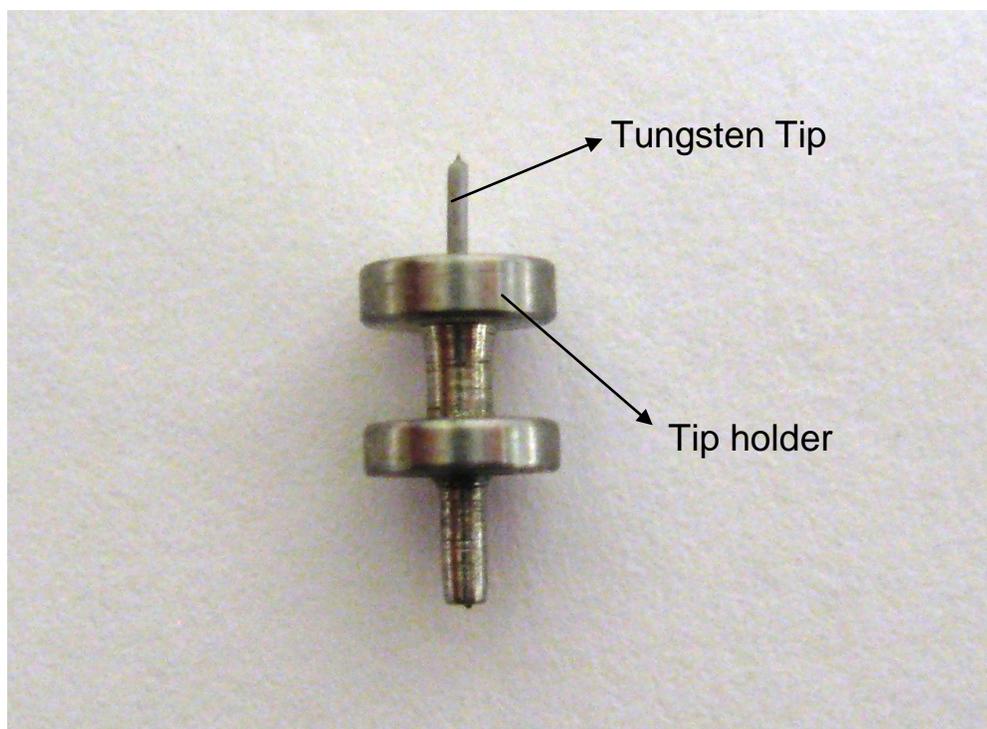


Fig. 2: Tungsten tip mounted onto the tip holder

## Steps:

1. ***Put gloves on;***
2. ***Clean all beakers and tweezers you plan on using*** - usually the tweezers are continuously stored in a beaker which contains Isopropanol;
3. ***Cut a fresh piece of the flexible tubing*** - the length of the tubing should be chosen such that it completely covers the part of the tungsten wire which will become the actual tip; also try not to make it flat on both sides (see the zoom in on the tubing in Fig. 3), so that you can recognize the part with the 'tip of the tip' when the etching process is over;
4. ***Cut a fresh piece of wire*** - it is better to cut a longer wire (~2'') since it has been observed that a higher ratio of exposed wire length to tube length prevents the tip from becoming elongated when the etching process is over;
5. ***Prepare the NaOH solution*** - weigh 16g of NaOH grains using the electronic scale; fill a clean beaker with 200 ml distilled water and pour the grains in; put the beaker into the ultrasonicator for ~ 10 minutes to allow the grains to dissolve (use a glass stirring rod if they did not dissolved completely after the 10 minutes have passed); finally, transfer the freshly prepared solution into a beaker similar to the one in Fig. 3;
6. ***Bend one end of the wire*** and make sure it is properly mounted onto the holder (a proper mounting implies no movement of the wire); ***remove the wire from the holder;***
7. ***Insert the bended end of the wire into the tubing;***
8. ***Mount the wire on the mini chuck*** as in Fig. 3

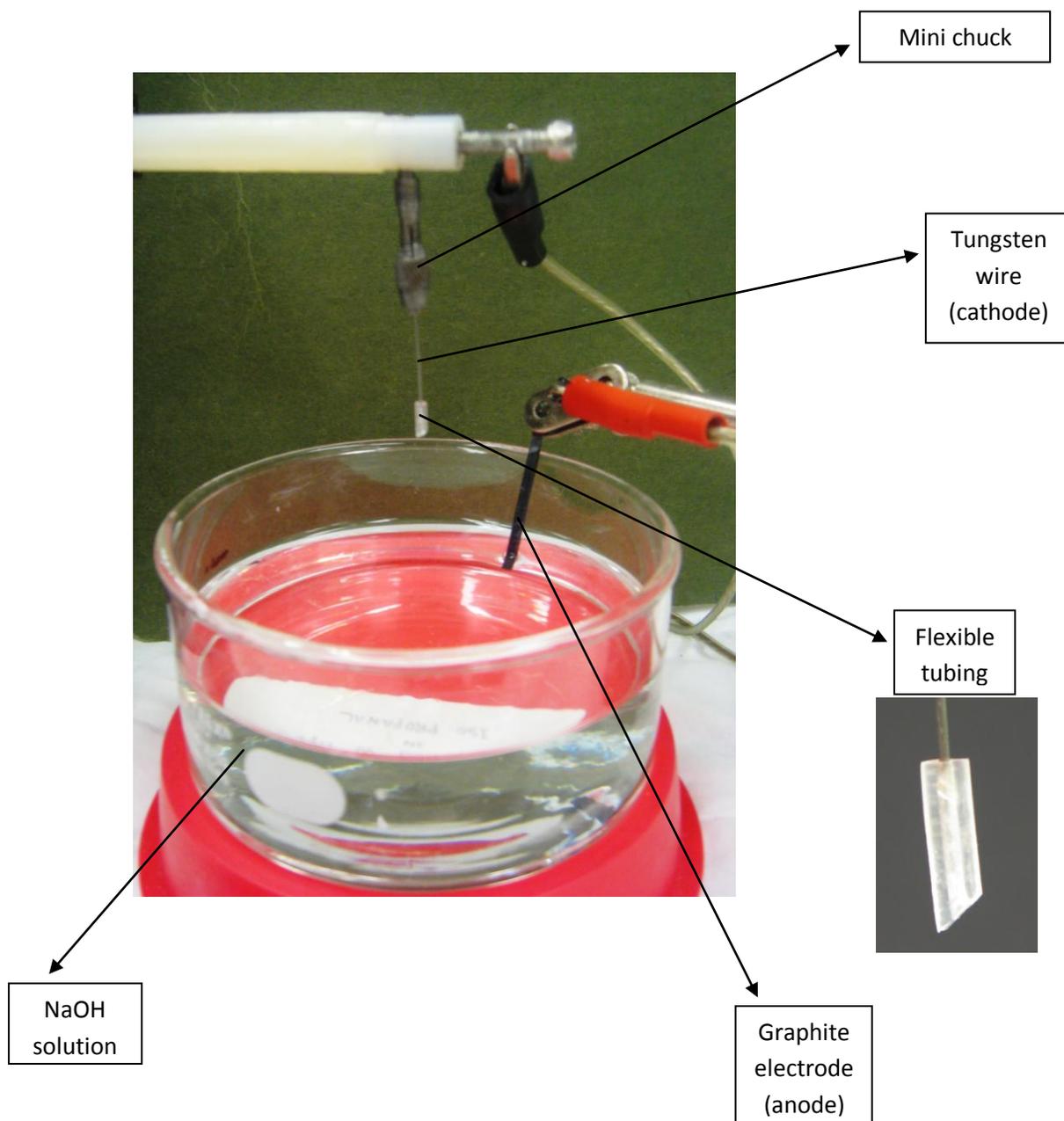


Fig. 3: Picture of the etching station used in preparing the tips

9. **Turn the power supply on** and **adjust** the AC voltage between the two electrodes (as mentioned before, the voltage should be  $\sim 4\text{V}$ ); **measure** the voltage using the multimeter; **turn the power supply off** ;

10. ***Submerge the part with the tubing into the solution***, making sure that the upper (flat) part of the tubing is very close to the surface of the solution (this will help achieving a tip that comes to a point rapidly);
11. Once you think that the upper part of the tubing is close enough to the surface of the solution, ***turn the power supply back on*** ; the etching process should begin;
12. Once the tubing (which contains the actual tip) falls off, ***carefully take it out of the solution*** with tweezers; using a piece of Tungsten wire, push the tip half way out and grab it with another pair of tweezers (see Fig. 4 and Fig. 5); the cleaning process should start immediately;

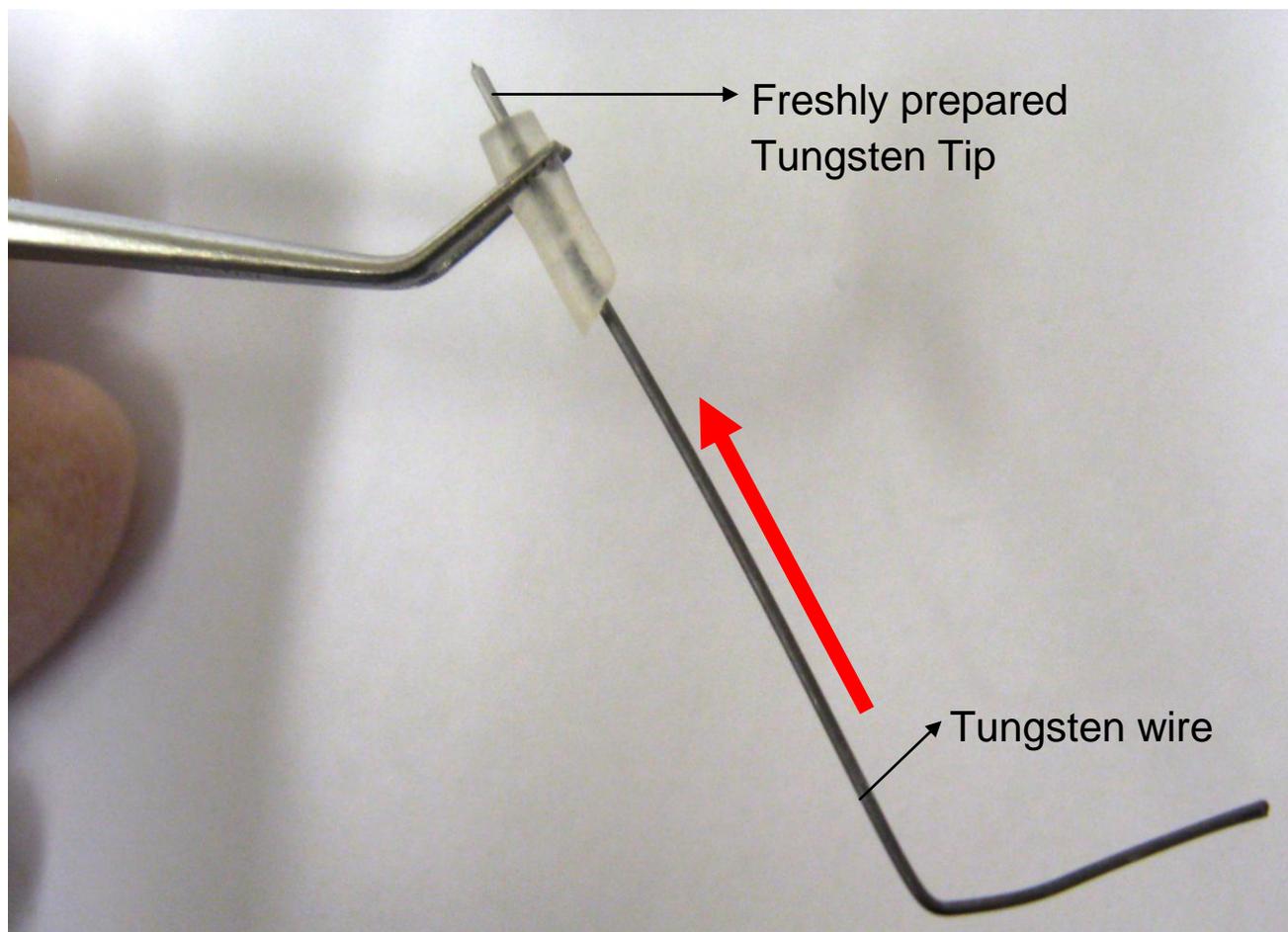


Fig. 4: Picture illustrating how the freshly prepared tip is pushed out of the flexible tubing, by making use of another Tungsten wire

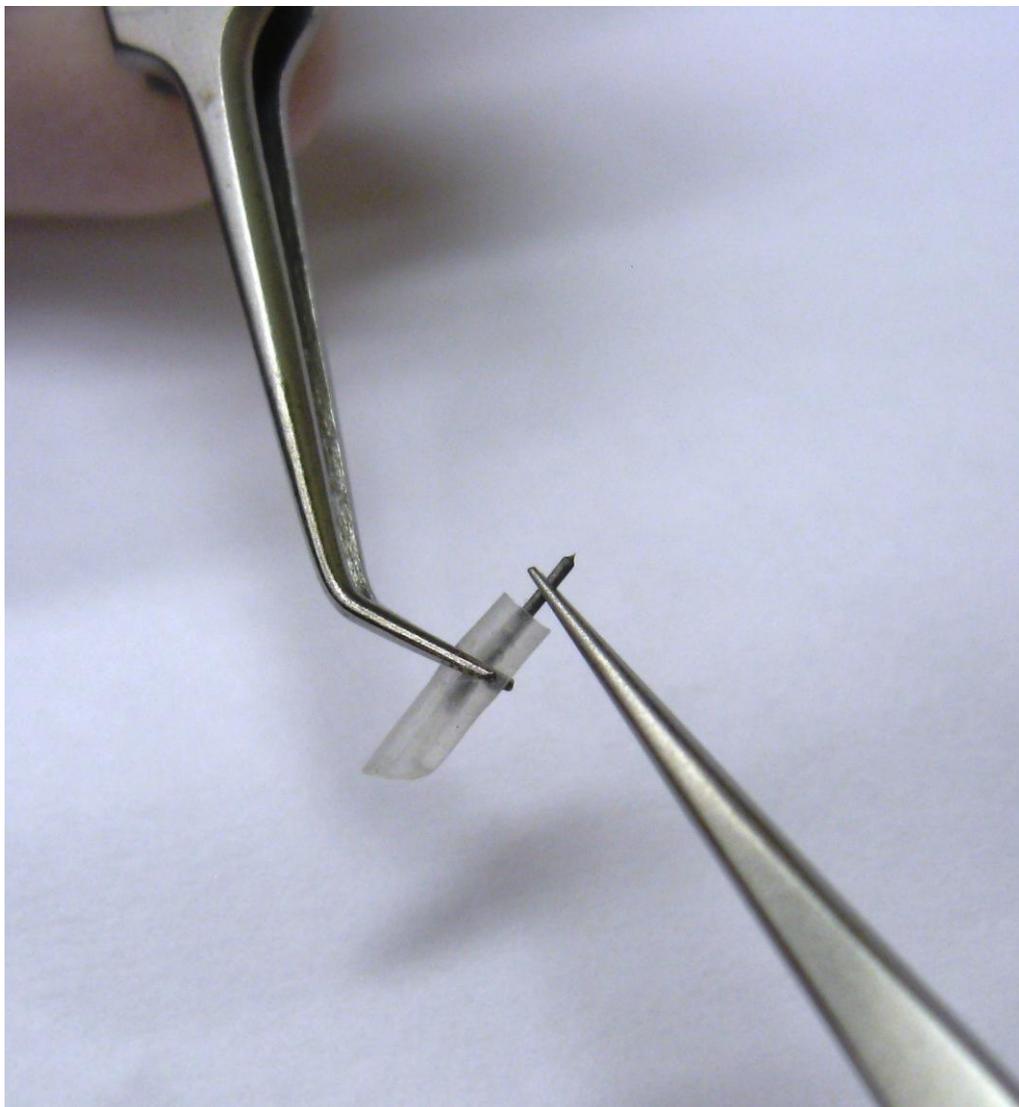


Fig. 5: Taking the Tungsten wire out of the tubing right before the cleaning process begins

13. ***While holding the freshly prepared tip with tweezers, clean it in distilled water and then in Isopropanol*** (using the ultrasonicator, for about 1 minute) as shown in Fig. 6; ***make sure to prevent the 'tip of the tip' from touching any surface*** (this can be checked by using the optical microscope - see Fig. 7 for a comparison between a tip that did not touch any surface and one that did);

14. After using the microscope, *clean the tip again in Isopropanol* following the same procedure as in the previous step;
15. *Carefully mount the tip onto the holder* (Fig. 2) and *make sure it is fixed in place*.

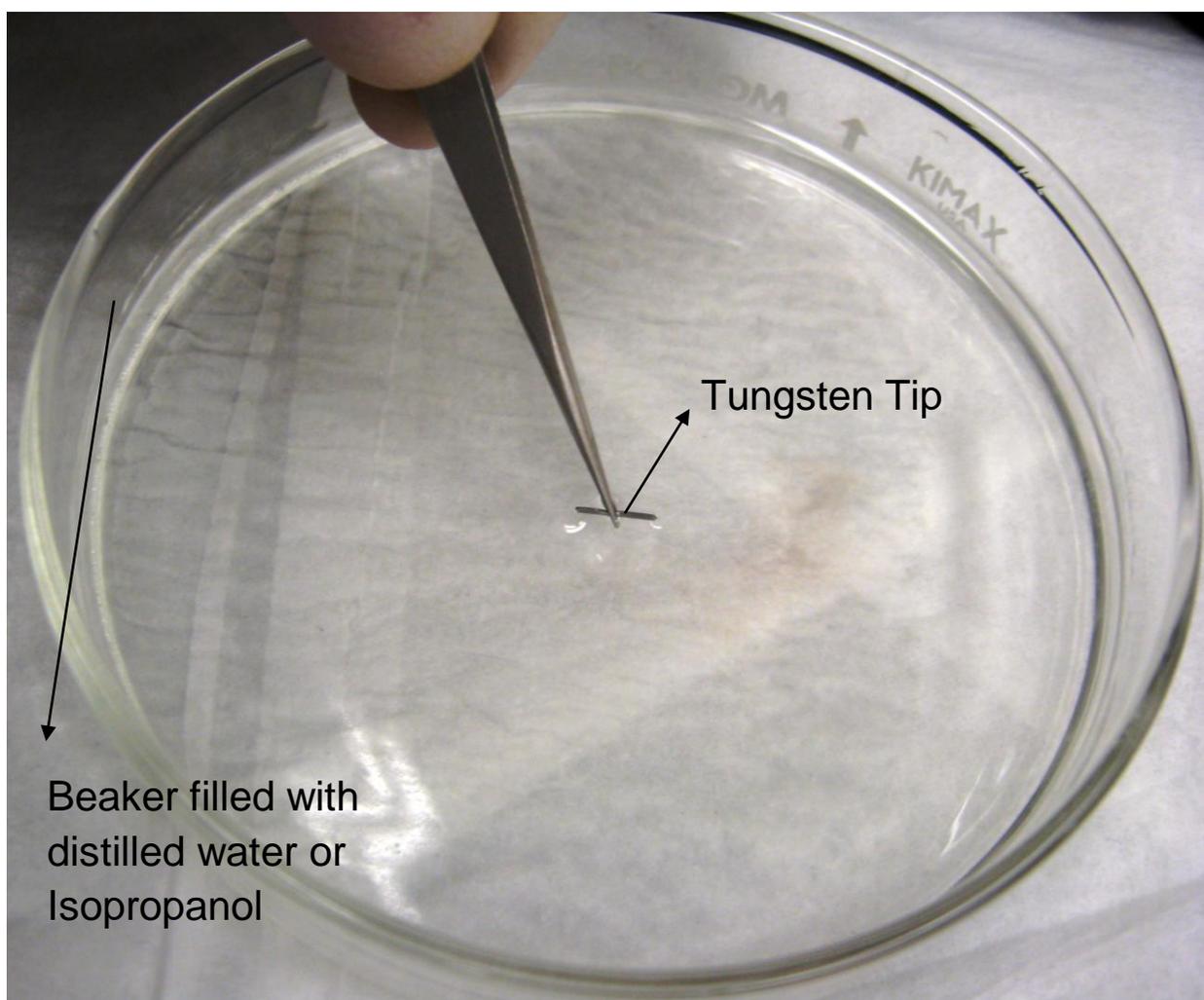


Fig. 6: Cleaning of the tip in distilled water and in Isopropanol; the beaker should be used inside the ultrasonicator

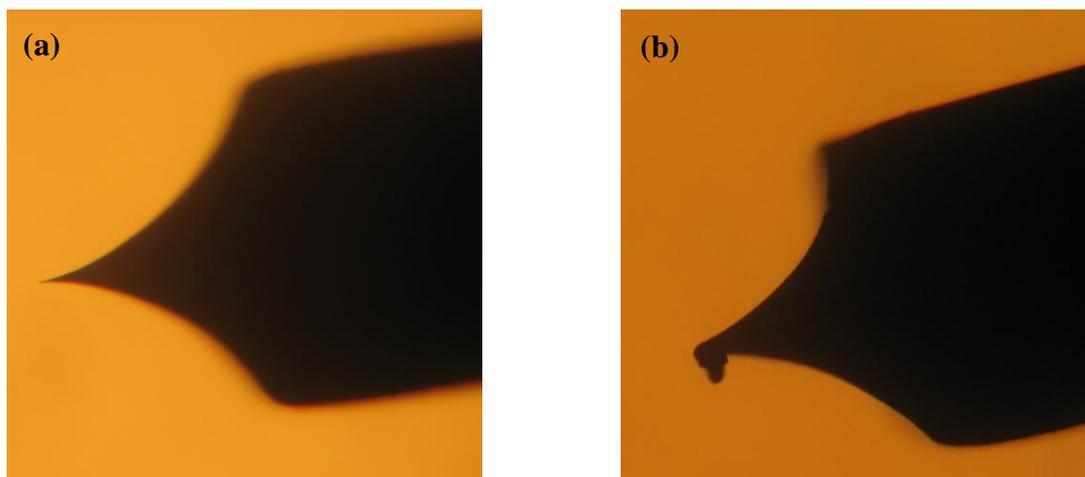


Fig. 7: Picture of the optical microscope image showing (a) a tip which did not come in contact with any surface and (b) one that did

Note: Due to the high degree of oxidation of Tungsten, it is advisable to *transfer the freshly prepared tips into the chamber as soon as possible.*