Versatile apparatus for etching scanning tunneling microscope tips

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We have developed an apparatus for easy and consistent etching of small tips suitable for use with a scanning tunneling microscope. Its unique features are free access to the etching region and a continuous supply of electrolyte for the production of many tips in succession.

An etching technique for scanning tunneling microscope tips described by Bryant *et al.*[1] has three noteworthy aspects: (1) the etching region is local, allowing controlled tip shaping; (2) the pinnacle is strained as it pulls away under its own weight, increasing the likelihood of an atomic sized asperity; and (3) the tip falls away from both the electrolyte and voltage source, automatically halting the electropolishing when it is done. These are accomplished by suspending the wire to be etched through the surface of the electrolyte into an air pocket trapped by surface tension inside of a small submerged glass tube.

We have improved upon this technique by eliminating several drawbacks which we found with this procedure. First, we found that the trapped air region is difficult to set up and maintain. Gases evolved during the etching would expand the volume considerably, requiring constant adjustment of the surface positions. Second, observation of the etching region was impeded by the physical arrangement of the apparatus. Third, recovery of the tip from the small tube was difficult in that it requires removing the catcher tube and disturbing the setup. Finally, in our attempts to form shorter tips from smaller diameter wire, the tip would often become overpowered by surface tension and flop up into the surface of the bubble instead of falling down free. This is due to the considerably reduced weight.

The improved apparatus is outlined in Fig. 1. The etching solution is suspended in a 3-mm-diam hole through a 1-mm-thick copper plate (a) by its surface tension. This plate serves as both the counterelectrode for the etching potential and a flow channel for electrolyte. Smaller, 2-mm-diam holes flanking the center hole are connected to the same by $\approx$0.5-mm-wide slots cut by hand with a jeweler's saw. Two 1-mm-o.d., 0.45-mm-i.d. tubes leading to the smaller holes provide a source and sink, respectively, for a continuous supply of fresh electrolyte. The tubes are sliced diagonally and inserted as in (b) to allow a stable capillarity bridge to the channels. The wire to be etched pierces the center of the larger hole, acting as the other electrode (c). The wire can thus be etched in a narrow region, the width of which is limited by the capillary forces of the surfaces.

The obvious advantage of this arrangement is the open access to both above and below the etch region. Many tips can be made in succession simply by feeding wire in from the top and catching the tips below with no limit on the length of wire protruding from either side. Clear views of both sides are possible.

Some details are as follows. It is useful to draw a wax outline around all edges of the copper cutout with a wax pencil. This helps keep the electrolyte from creeping across the copper and changing the geometry of the surface. It is important to keep all wetted surfaces to a minimum so that increases in the trapped volume increase the capillarity pressure as rapidly as possible. Smaller wetted areas force small surface curvatures when the volume deviates from equilibrium.

The flow equilibrium is determined by the relative positions of the supply reservoir, the waste reservoir, and the capillary electrode. The overall flow rate is proportional to the level difference between the two reservoirs and the equilibrium capillarity curvature can be adjusted by moving the reservoirs together with respect to the electrode. As the reservoirs are lowered, the curvature becomes concave until the surfaces in the center electrode contact each other, risking an opening up of the surface. As the reser-

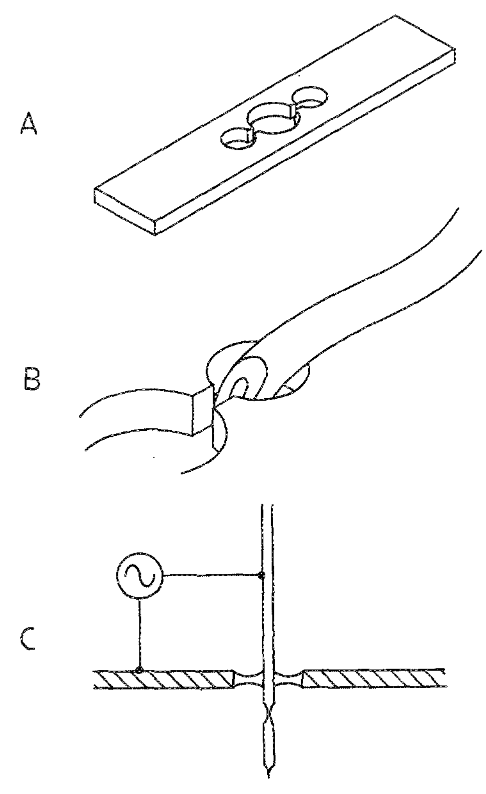

FIG. 1. Schematic drawings of the electrode arrangement. Counterelectrode plate (A). Detail of tube junction to side hole (B). The wire to be etched passes through the center hole with the electrolyte wicking between the electrodes (C).

voirs are raised, the curvature becomes increasingly convex, ultimately forming a drip. There is a reasonable range of curvature for which the equilibrium is stable for ≈ 1 cm variations in either reservoir height. Slightly concave surfaces are favorable.

The electrode and wire can be held by any fixed clamping arrangement. To start the flow, it is easiest to first adjust the reservoirs ("labjacks" with beakers are perfect) so that both free surfaces are level with the electrode. The reservoirs can then be slightly pressurized one at a time so that the electrolyte flows down the tube and wets the electrode. The liquid can then be drawn across the 3-mm hole with a wetted probe. The supply reservoir is then raised about 5 cm and the waste reservoir is lowered a similar distance, then adjusted to give the desired curvature. With ≈ 30-cm source and drain tubes, this configuration results in a flow of about 7 ml/h.

A flow rate of 1 ml/h at a typical concentration of 4 wt. % KOH used for tungsten is equivalent to an ion circulation of approximately 20 mA through the etching region. The electrical currents during etching are usually smaller than this, much smaller just before the tip is etched through. The flow rates are therefore sufficient to keep the relatively small active electrolyte volume supplied with fresh solution. The electrode characteristics are roughly such that the current is proportional to the square of the ac voltage with 1-Vrms electrode potential resulting in a 5-mA current with 0.2-mm wire.

Tip shapes can be controlled by etching speed, moving the wire with respect to the etch region, or the ac frequency.[1,2] The continuous supply of fresh electrolyte helps assure reproducibility. For very small tips (we have experimented with wire down to 0.08 mm) we have found it useful to etch a thin neck below the tip [Fig. 1(c)] leaving several millimeters of wire weakly attached to its back end. This section can be used to handle the much shorter tip, breaking it off before use. It also allows the straining force applied during the tip etching to be adjusted independent of the tip size since this additional mass can be arbitrarily large. This solved a problem we had of very small tips occasionaly being sucked into the electrolyte meniscus.

Finally, we are able to produce many tips in succession since the finished tips fall out underneath, and a new length of wire can be fed into the top. The length of wire used as a handle allows the finished tips to be caught reliably in a cup, in our case one of many small blind holes drilled in a piece of plastic to make a rack. This can be repeated indefinitely as the electrolyte is always fresh.

[1] P. J. Bryant, H. S. Kim, Y. C. Zheng, and R. Yang, Rev. Sci. Instrum. **58**, 1115 (1987).
[2] L. A. Nagahara, T. Thundat, and S. M. Lindsay, Rev. Sci. Instrum. **60**, 3128 (1989).