

Preparation and characterization of electrochemically etched W tips for STM

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Abstract. We have investigated methods for cleaning dc-etched polycrystalline tungsten tips for scanning tunnelling microscopy (STM). The cleaning methods include Ar-ion sputtering, heating, chemical treatments and Ne-ion self-sputtering. We correlate transmission electron microscopy images of the tip, field-emission data from the tip and images of a clean Cu(111) surface to find an optimum procedure for STM imaging. Clean and sharp tips are made by sputtering, combined with careful heating by electron bombardment. We found that optimum sputtering was obtained either by use of a 4 keV Ar-ion gun for a few seconds or by self-sputtering with Ne ions for a few seconds or until decapitation occurs.

Keywords: STM tip shape, STM tip composition, UHV, TEM, STM, scanning tunnelling microscopy

1. Introduction

The size, shape and cleanliness of a scanning tunnelling microscope (STM) tip are very important for the resolution of a STM. Tips not properly prepared might have several minitips, of which the one closest to the surface gives the image. If there are several tips, the tip from which electrons tunnel might change when the tip is scanned. This gives double or even multiple imaging of features at the surface. If the tip is not free of contamination and oxide, the tunnel junction may be unstable and cause irregularities in STM imaging and scanning tunnelling spectroscopy (STS). The tip therefore has to be prepared carefully. A Pt–Ir tip used in air on atomically flat surfaces can often be made to work properly just by cutting it with a pair of scissors. For UHV W tips are frequently used. A W tip has to be prepared by electrochemical etching, because of the hardness of the material. Possibly there are as many tip-preparation procedures as there are people making tips. Some publications, but far from all, report results from scanning electron microscopy (SEM) and transmission electron microscopy (TEM) images. It is possible just to etch the tip and rinse it in de-ionized water, but many procedures to optimize the tip performance have been suggested. One large problem with tungsten tips is that often they are contaminated during the etching procedure. The contaminants consist of H₂O, CO, H₂, O₂, hydrocarbons [1, 2], etching residuals (KOH, NaOH) and tungsten oxide, WO₃. These oxides and contaminants cause unstable tunnel currents and the tip loses

some of its metallic behaviour, which can result in tip crashes and thereby damage to the tip and the surface. The tungsten oxide layer seems to be a few nanometres up to about 20 nm thick with no exact correlation to the preparation procedure used. Ibe *et al* [3] conclude that a low etching voltage gives less oxide than does a high voltage. Many methods to remove surface oxides and contaminants have been proposed, for example sputtering [4, 5], annealing [4, 6, 7], exposure to a well-focused laser [8], applying a low reverse voltage [9, 10], normal (tip oriented downwards) and reverse (tip oriented upwards) etching [11] and chemical cleaning with HF [12] which dissolves WO₃ but not W. Annealing and sputtering are the standard procedures for tip cleaning. Annealing is mostly done by electron bombardment inside the UHV chamber. Sputtering is used with a variety of parameters both for cleaning and for sharpening.

A method of determining the sharpness of the tip is to measure the field-emission current from the tip by applying a high voltage between the negatively biased tip and another electrode [13]. A sharp tip will give a large field-emission current, whereas a blunt tip will give a small current. Except for the obvious test of STM tips, i.e. taking pictures of the surface of interest, it is possible to look at them in microscopes, optical as well as SEM and TEM. An optical microscope can be used to tell whether the tip is bent on a large scale, while SEM and TEM images give information about the size and shape of the tip. From TEM images one can also observe whether there is an insulating layer, for example an oxide, on the tip and in that case obtain an estimation

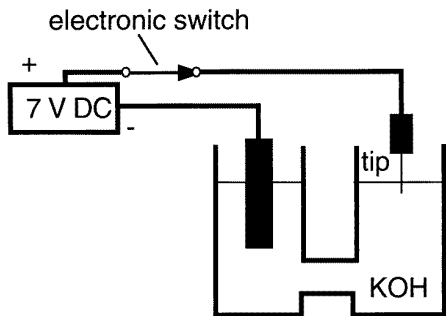


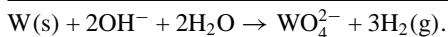
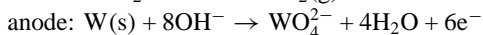
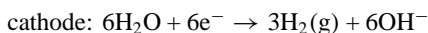
Figure 1. The experimental tip-etching configuration. In the electrochemical cell a W wire is used as the anode and a stainless steel rod is used as the cathode. The two electrodes are placed in separate beakers to avoid the bubbles developing from H_2 at the cathode disturbing the etching.

of the size of the insulating layer. TEM studies have been performed by various groups [4, 5, 9, 14, 15]. Chemical studies of various tip materials have been performed by for example x-ray photoelectron spectroscopy [2], Auger electron spectroscopy [2, 16] and microscopy [7, 17] and energy dispersive x-ray analysis (EDX) [18]. Scanning tunnelling spectroscopy (STS) data are frequently used as an indicator of the presence of oxide on the tip. If the tip is oxidized the STS data should not reveal a metallic tunnel junction, i.e. the $I-V$ curve would not be linear, but should have a gap-like structure around zero bias voltage. However, authors of only a few studies have correlated preparation techniques to microscopic tip shape, field-emission data and actual STM imaging, which we do in this study. We have studied dc-etched W tips and tried to correlate TEM images, field-emission data and STM/STS data of a clean Cu(111) surface to find the optimum scanning tunnelling microscopy tip. We have used many different techniques of tip preparation including sputtering, heating, ac polishing and chemical treatment. It is important to note that the exact atomic geometry of the tip is important for tunnelling and STM imaging and that this shape often changes during tunnelling. In this investigation we are thus not able to correlate a given tip microscopic shape or tip preparation to a certain image quality. We can, however, correlate different preparation techniques to drastic changes in the probability of acquiring good STM data.

2. Experimental details

2.1. Etching

For etching we used a tungsten wire as the working (anode) electrode in an electrochemical cell and a piece of stainless steel was taken as the counter electrode (cathode) (figure 1). A 2 M KOH solution is used as the electrolyte. The following reactions take place [6]:



To avoid disturbance at the anode from the H_2 gas evolving at the cathode the two electrodes were placed in separate beakers connected by a glass tube.

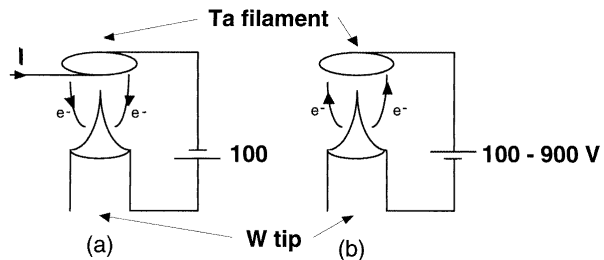


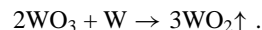
Figure 2. The heating procedure (a). A current of about 1 A is sent through a 0.1 mm Ta filament formed as a circle. When the tip is biased positively, electrons will flow to the tip and heat the tip apex. The field-emission procedure (b). If the tip is sharp enough and the polarity is reversed, field-emitted electrons will flow to the filament.

The electronics consists of a constant-voltage power supply with an automatic switch-off control [3, 19]. During etching the current through the cell will decrease linearly with time because the resistance of the cell increases when the area of the wire in the electrolyte decreases. The wire eventually breaks at a neck that is formed during etching, since the rate is enhanced in a region just below the KOH surface. In our cell the wires broke after about 20 min, and the current quickly fell below a pre-set limit, below which the supply is automatically cut off by the electronic circuit. Across the cell there was a $0.47 \mu F$ capacitance to allow smoothing of the tip after the break, enhancing the reproducibility.

Apart from KOH the solution contains a few drops of 2% Decon added to lower the surface tension. After each etching step the tips were rinsed in four subsequent beakers of hot de-ionized water and methanol. The tips were cut from straight W wire of 0.38 mm diameter (Goodfellow Ltd, England) and were ultrasonically cleaned in 2% Decon. The wires were then slightly electropolished over a large part of the wire. In this step, six wires at a time were etched. Because the cutting of a tungsten wire introduces defects, the damaged material near the cut was removed by etching away about 4 mm of the wires. The final etching starts with electropolishing over a large area of a single wire. The tip was then rinsed and lowered to about 1.5 mm below the KOH surface and was moved 0.01 mm further down halfway through the etching to optimize the neck formation [3]. The current through the cell was measured with a HP 34401A multimeter connected over a GPIB interface and controlled by a LabVIEW program to directly observe any irregularities in the etching current. Finally the tip was mechanically cut to fit the tip holder of the microscope.

2.2. Cleaning

Heating was made inside our UHV STM chamber (Omicron Vakuumphysik GmbH, Germany) with a standard heating procedure (see for example Chen [6]). We applied 100 V between the tip and a 0.1 mm diameter Ta filament. The filament was formed as a circle of diameter 3 mm, figure 2. With a current of about 1.0 A through the filament, thermally emitted electrons flow to the tip and heat it. The WO_3 on the tip will then react with W according to [6]



WO₂ sublimates at 800 °C, in contrast to W that has a melting point of 3410 °C, which suggests that there will be no blunting of the tip as the WO₂ sublimates. We heat the tips for 30 s at a time, with a maximum current to the tip of 0.1 μA for our heating configuration.

Sputtering was performed with an Ar-ion sputtering gun at energies 0.5–4 keV for durations from a few seconds up to 10 min at a pressure of about 1.5×10^{-5} mbar. Self-sputtering is performed in the heating configuration, using the filament as the counter electrode, at a gas pressure (Ne or Ar) of about 1.5×10^{-5} mbar. When a high negative voltage is applied to the tip, field-emitted electrons will ionize the noble gas and create positive ions that are accelerated towards the tip. Field-emission currents depend on the tip sharpness and on the distance between the tip and the filament. Two different approaches are used when self-sputtering, the first is to decapitate the tip and the second is to clean the tip. In the first method we first try to achieve field emission of about 50 μA, this is normally done by the sputtering process since the current usually rises as the sputtering proceeds, but adjustment of the tip position (i.e. moving closer to the filament) may be needed in order to achieve high enough currents. After reaching about 50 μA (usually at a bias voltage of around 800 V) we try to keep the current at about 10–50 μA by gradually lowering the applied voltage in steps of 50 V. This procedure is continued until there is a sudden increase in current which signifies that the decapitation has occurred. The voltage is then automatically switched off. The other approach is to sputter for a few seconds to clean the tip. Chemical treatment was performed by dipping the tip into 47% hydrofluoric acid (HF) solution for 3 min and then rinsing in de-ionized water. Ac polishing was performed in a 10% solution of a commercial developer (neutol) for 1–3 s at a frequency of 400 kHz. We tried various frequencies and noted that some frequencies tended to leave a black layer on the tip. We therefore chose a frequency for which no residuals were visible.

2.3. Characterization methods

We investigated our tips in a JEOL 2000 FX TEM. For large-scale features a JEOL JSM-6301F SEM was used for imaging. To characterize the tip shape and cleanliness we measured the field-emission current between the tip and the heating filament. The tip was then biased negatively with respect to the filament, but in the same geometry as that used for heating. We also tried field emission to a gold surface instead of to the filament and the results did not differ, i.e., we got the same currents for the same applied voltages. Finally, we measured STM images and STS spectra of a clean Cu(111) surface to correlate tip preparation and image quality.

3. Results

We investigated tips prepared in different ways, both tips that were not cleaned at all and tips prepared by one or a combination of the different procedures described above.

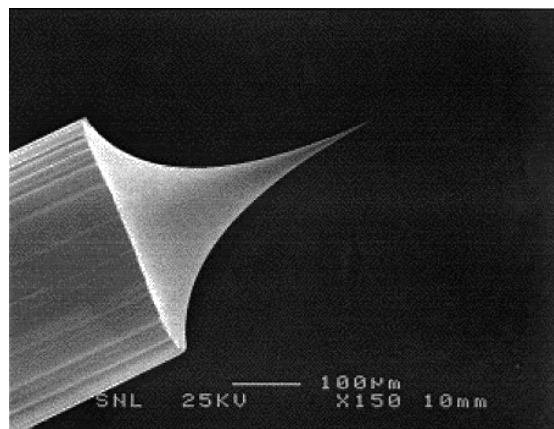


Figure 3. A large-scale SEM image of a dc-etched W tip. The tips all look reproducible at this scale, but it is not possible to tell a sharp tip from a blunt one within this resolution.

3.1. Field emission

Molten or mechanically damaged tips, as well as tips blunt from the start, exhibited no field emission for voltages up to 900 V. This is a simple way to investigate whether a tip has crashed. Sharp tips, untreated, exhibited field emission and the emission usually increased when the tips were heated. For some tips the emission currents were stable; for others they were unstable, probably due to rearrangement of the atoms at the tip apex. ‘Good’ tips gave emission currents of a few tenths of an nano-ampere for a negative tip bias of 300 V. The emission current was very dependent on the distance between the tip and the filament, but the behaviour was the same, although the actual *value* of the current might differ. When the tips were accidentally crashed or melted, the tips exhibited no field emission for voltages up to 900 V. This proves that the emission really does come from the tip rather than from the relatively sharp edges where the etching neck was formed. In our experimental configuration it is not relevant to compare the exact values of field emission from different tips, since the actual values are dependent both on the exact tip–filament distance and on the atomic geometry, which we cannot control. We therefore only use field-emission data qualitatively.

3.2. TEM images

3.2.1. Untreated tips. Untreated tips were etched by the above etching procedure and checked in SEM and TEM (figure 3 and 4). Both tips just etched (40 min before imaging) and 1-day-old tips (kept in air) were investigated. They had a diameter of about 10 nm and an insulating layer of thickness about 5–10 nm, as reported earlier [5, 16]. This layer probably consists of WO₃ and KOH residuals. A crude chemical analysis with EDX showed that at least W was present. There were no distinguishable differences between the 1-day-old tips and the just etched ones. Inside the insulating layer there were sharp tip shapes. Sometimes the insulating layer followed the tip shape, but also strange, irregular shapes were detected. It was impossible from the SEM images to conclude more than that the tips are reproducible on a ‘macroscopic’ scale. It is not possible to

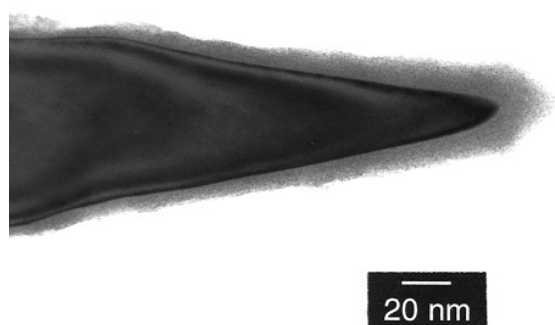


Figure 4. A TEM image of an untreated electrochemically etched tip. The tip apex diameter is less than 10 nm. Outside the sharp tip shape there is an insulating layer, about 5–10 nm thick. A crude chemical analysis shows that at least W is present in the insulating layer, so that the layer probably consists of WO_3 .

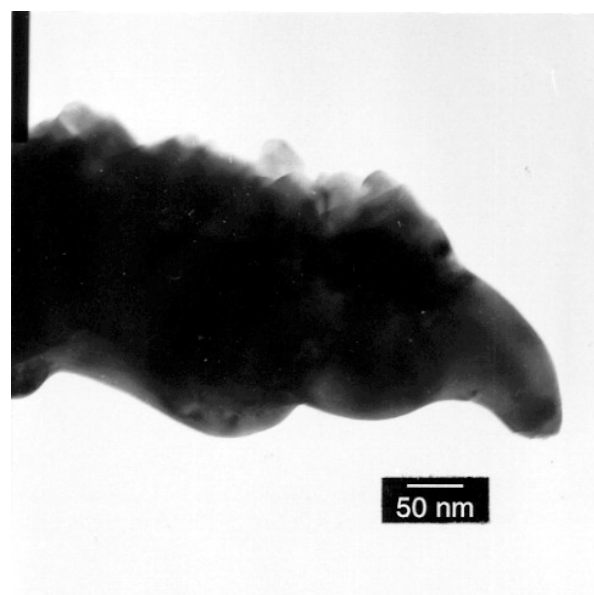


Figure 5. A tip heated by electron bombardment to a maximum of $1.0 \mu\text{A}$ and therefore somewhat melted. The very end of the tip is clean, while there is still an insulating layer further down.

distinguish a sharp tip from a blunt one with the resolution of the SEM.

3.2.2. HF-treated tips. The HF-treated tips had the same shape as the untreated ones, indicating that the HF does not remove W. Tips treated with 47% HF exhibit a contamination layer thinner than that on the untreated ones, but there are still signs of an insulating layer. Overall this preparation did not seem to work as expected, so we did not proceed with this method.

3.2.3. Heated tips. The TEM images of heated tips showed that the heating was very local. Only the uppermost part of the tips was clean, while the rest still had contamination. We also noted that it was easy to melt the tips accidentally. In our heating geometry the tip melts at around $0.5 \mu\text{A}$, whereas

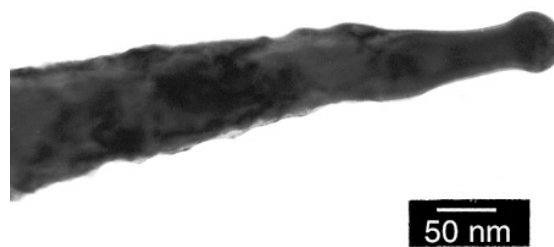


Figure 6. A tip sputtered at 4 keV for 10 min. The tip is clean over a large area. At the tip end a neck is forming because of the sputtering process. The tip apex has a diameter of about 50 nm.

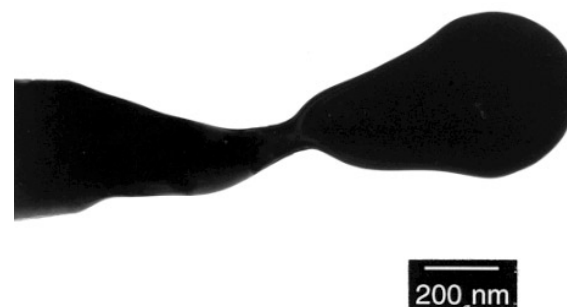


Figure 7. A tip sputtered with 4 keV for 30 s. The tip is clean and has a thin neck and a large 'head'. The diameter of the head is about 200 nm. This tip does not produce any field-emission current.

on placing the tip inside two turns of a filament (with a total of five turns), the current applied to the tip can be up to 0.5 mA without melting the tips, which was verified by field-emission measurements. By melting we mean that the field emission disappears. However, almost all tips are affected by the heating, in such a way that the formation of a neck is started by the local heating. Figure 5 shows a heated tip that is molten at the very end.

3.2.4. Ac-polishing. The ac-polished tips were extremely blunt and highly contaminated. We could not tell whether or not this treatment removed any oxide and did not proceed with this method.

3.2.5. Ion-gun-sputtered tips. Tips ion-gun sputtered for 30 s to 10 min exhibited a large clean area at the tip end. However, the TEM images showed that a neck was formed. The shape of the neck varied from a wide neck with a ball at the end (figure 6) to a thin neck with a large and strangely shaped tip apex (figure 7). Tips sputtered with a higher energy looked cleaner and smoother than did tips sputtered with a lower energy. Since it seemed that sputtering removed not only oxide but also W, the sputtering of oxide should be fast. A few seconds of sputtering sufficed remove oxide, while avoiding neck formation.

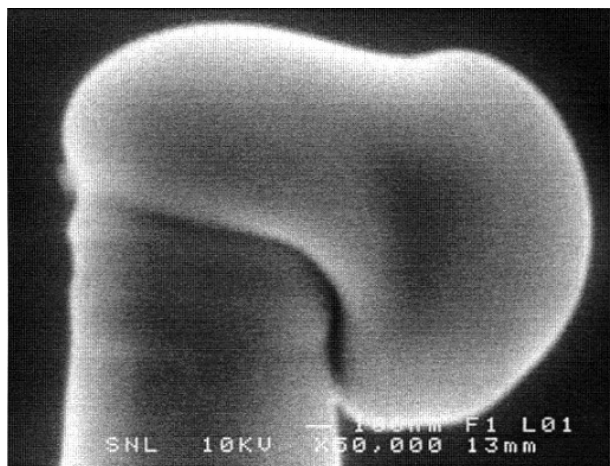


Figure 8. A large-scale SEM image of a tip that suddenly lost all of its field emission during self-sputtering.

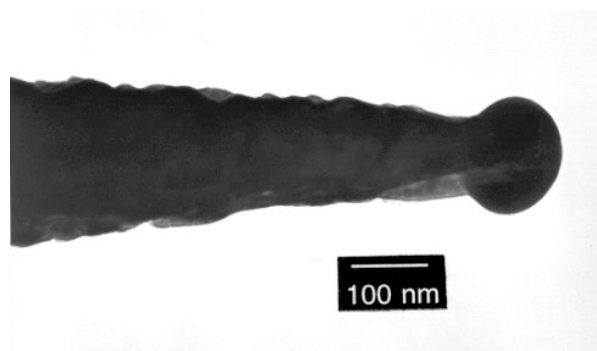


Figure 9. A tip decapitated during self-sputtering. The neck is probably formed by the heating procedure. The diameter of the apex is about 100 nm.

3.2.6. Self-sputtered tips. Self-sputtered tips were clean over a large area, as are the ion-gun-sputtered tips. In our experimental procedure, we can measure the current to the tip and use this for monitoring the sputtering process. We observed two distinct features during sputtering. First, the field emission sometimes suddenly dropped to zero. Figure 8 shows a SEM image of a tip with this behaviour. The tip is quite blunt and has a molten-like shape. We have occasionally observed flashes going between the tip and the filament in connection with the sudden drop in field-emission current. Second, the field emission suddenly increased by at least $50 \mu\text{A}$ from a normal value of tenths of a micro-ampere. We interpreted this as decapitation of the tip, which had been observed earlier by Schiller *et al* [20]. Decapitation occurs when the neck formed breaks and a very sharp tip is left at the apex. Since the sputtering had been stopped, the neck shown in figure 9 was probably formed by the heating following the self-sputtering. The reason for heating is to repair defects induced by sputtering which can be seen below the heated part at the outermost tip [20].

3.3. Imaging and spectroscopy

To test the quality of tips in STM we imaged a clean single-crystalline Cu(111) surface. With untreated or molten tips we

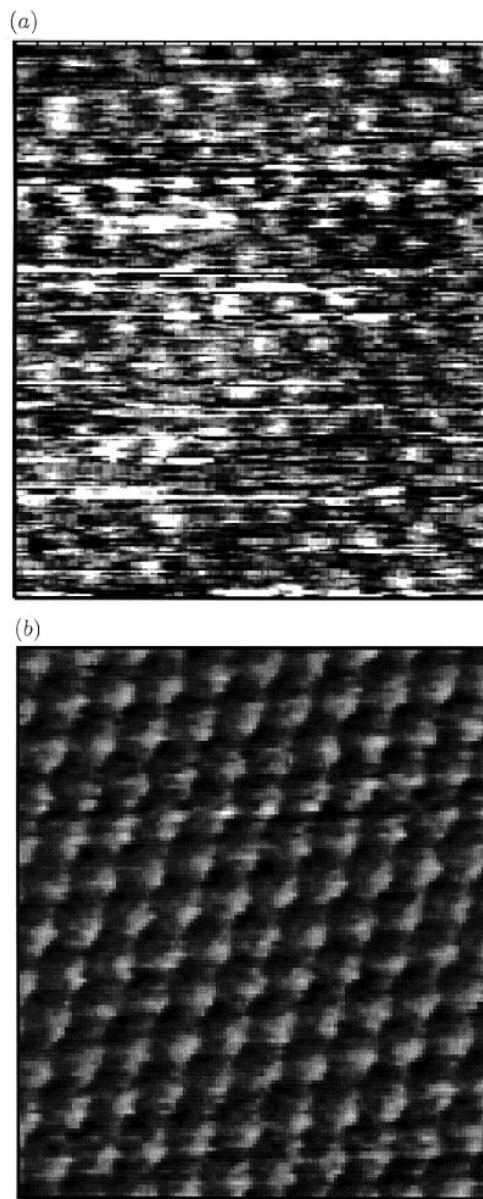


Figure 10. An atomically resolved Cu(111) surface. (a) A molten tip with $U = -20 \text{ mV}$ and $I = 4.1 \text{ nA}$. Atoms are clearly visible, but the image is noisy. The image size is about $2.2 \text{ nm} \times 2.5 \text{ nm}$. (b) A tip prepared by sputtering at 4 keV for 5 s and heating to $0.08 \mu\text{A}$. $U = -310 \text{ mV}$ and $I = 0.47 \text{ A}$. The image size is about $2.5 \text{ nm} \times 3.0 \text{ nm}$. No processing of images was performed except for correction for distortion.

occasionally got atomic resolution, but the images were noisy and it was often impossible to get low-noise and reproducible tunnelling spectra. On a larger scale, the images exhibited expected step heights and structures. We often got low-noise atomic-resolution images from tips that after modest, repeated heating exhibited high field emission. Also the spectroscopic results had low noise. Tips that had been ion-gun-sputtered for a long time provided unstable STM images. High noise and large spikes, combined with a tendency to give oscillating currents, were found with almost all these tips. With a drastically reduced sputtering time, a few seconds, the tips became more stable, giving STM

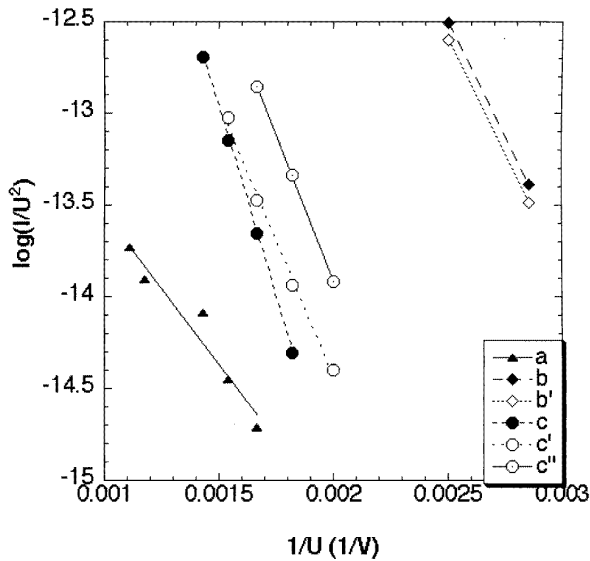


Figure 11. Field-emission data before and after heating and decapitation. A good tip exhibits field emission with a smaller slope and the curve is shifted to the right-hand side, i.e. current flows for a smaller applied voltage. Field emission from a tip (a) after 3 s sputtering, heating and subsequent scanning of the Cu(111) surface, (b) after decapitation and (b') after heating, (c) after scanning the Cu(111) surface, (c') after heating and (c'') after another heating. (Linear fits have been made as guides to the eye.)

images comparable to the ones obtained from sharp tips which had been heated only. Self-sputtered tips occasionally exhibited the oscillating behaviour, but mostly gave STM data comparable to those from heated tips and tips that had been sputtered for a few seconds. Figure 10 shows a comparison of a typical atomic resolution image of Cu(111) with a molten tip and a tip prepared by sputtering with 4 keV for 5 s and heating to $0.08 \mu\text{A}$. The field-emission data showed that the tips usually changed during scanning. Both better and worse field emissions were found.

4. Discussion

Theoretically field-emission currents follow the Fowler–Nordheim equation for high applied bias voltages. Following the Fowler–Nordheim equation, the dependence of the field-emission current (I) on the applied bias voltage (U) and the radius of the tip (r) in the Fowler–Nordheim regime can be expressed by [21]

$$I \propto \left(\frac{U}{r}\right)^2 \exp\left(-\frac{\text{constant} \times r}{U}\right).$$

The formula shows that a smaller slope in $\ln(I/U^2)$ versus $1/U$ means a higher field and thereby a sharper tip. A shift to higher values of I/U also indicates high quality, since this means that a current flows for smaller applied voltages [13]. Our tips followed this behaviour when they were heated, as shown in figure 11. The increase in field-emission current after heating can be due either to cleaning, i.e. lowering the insulating barrier thickness, or to atomic rearrangement at the tip apex. One has to be very careful and stop when the field

emission changes substantially, in order to avoid melting. The tips that had been decapitated exhibited an extremely high field emission, with the current rapidly increasing with the voltage.

Chemical treatments did not work as we had expected. The ac polishing with neutol gave tips with much more contamination than untreated tips. These tips were also blunt, probably due to the fact that we did not just polish but also etched the tip. TEM images of tips prepared with backpolishing have been published by Fasth *et al* [9] and Garnæs *et al* [15] showing that there is no blunting of the tips. They backpolished the tips immediately, in the same etching bath, after the initial ac etching. The difference between their and our experimental procedure is that we need to remove the tip from the etching cell to change the etching solution. This might cause important differences making our procedure unsuccessful. Since we have sharp tips that can be cleaned by a different procedure, we did not go further with the ac-polishing investigation. There are no published TEM images of HF-treated tips. The only published work regarding HF gives STM and STS data on highly oriented pyrolytic graphite in air as an indicator of cleanliness [12]. Considering our experiences during this work, we do not believe STM/STS data to be reliable enough to allow one to judge the quality of the tips. Our TEM images of the HF-treated tips showed that HF might remove some of the oxide, but the treatment seems to be irreproducible. It should, however, be noted that our HF concentration is somewhat lower (47%) than that used by Hockett and Craeger [12] (51%). Dc-etched tips have a higher aspect ratio (radius divided by length) than do ac-etched tips. This might make tips mechanically unstable. However, we noted that the tips were robust with respect to dipping, boiling, cutting (to fit the tip holder in the microscope) and even dropping as long as the very end of the tip was not touched.

The shape and cleanliness of the STM tips seem to be very dependent on the exact conditions of cleaning. Heating by electron bombardment from a filament will remove oxide and contamination locally from the tip apex, but the geometry of the experimental apparatus is crucial for the exact heating. By checking the field-emission current from the tip to the filament it is possible to find the conditions for tip melting. Heating also rearranges the atoms at the tip apex, which can give a more (or less) stable tip. Heating can, for example, cause neck formation, as found by Drechsler *et al* [22], as we see in our TEM images. This neck can be more or less pronounced depending on the heating procedure. However, heating is too local to clean the tip sufficiently. Scholz *et al* [23] have designed a special experimental apparatus to focus the electron impact at the outermost tip. We do not believe this to be necessary, since the heating of the tip is always very local according to our TEM images. Contaminants might electromigrate along the tip surface during scanning, giving a contaminated tip apex even after cleaning. Sputtering cleans a larger part of the tip, which minimizes this effect. The formation of necks during sputtering is reported by Schiller *et al* [20]. They claim that this is due to three factors. Firstly, the density of impinging ions will be largest at a certain distance from the tip apex because of the favoured trajectories of the ions, secondly the sputtering yield is dependent on the

angle of incidence and thirdly sputtering is favoured at areas with a high step density. The neck formation will, according to Schiller *et al*, proceed in an oscillatory manner when the process starts again after decapitation. To prepare a tip with no neck one has to stop the sputtering when a neck which already has formed breaks, or before any neck is formed. A few seconds of sputtering sufficed to remove oxide, while still keeping the tip shape rather intact. We believe that the neck is the reason for the instability we found in STM measurements.

Since the heated tips also have a slight neck, one could argue that these also would be unstable. However, one has to be aware that the heating and sputtering types of neck formation are two different processes. During sputtering, W is removed from the neck by the impinging ions, whereas the neck formation during heating is due to a slight melting of the tip apex. The necks formed by heating should therefore be very smooth and uniform, whereas the sputtered ones possibly consist of many defective areas.

In the self-sputtering arrangement we can follow the etching process by monitoring the current. It is therefore possible to stop the sputtering when decapitation occurs and thereby obtain a sharp tip without a neck. Decapitation was found to occur when we first let the current increase to about tenths of a micro-ampere at 900 V. The tip then sharpens due to sputtering, giving a higher field-emission current. The rise in field emission continues until we get a sudden increase in field emission due to decapitation of the tip. When we were trying to stop the sputtering manually by switching off the supplies, we had problems with new necks forming, so we use an electronic cut-off control to automatically set the voltage to zero when the field-emission current suddenly increases as the neck breaks. We have no solidly grounded explanation for why some of the self-sputtered tips suddenly exhibit a drop in field emission and appear to become molten. We have, however, observed flashes, which can be attributed to conducting channels in the sputtering gas. The increase in current in such a channel may account for the molten shape of the tip. We occasionally found self-sputtered tips that consist of two or three different tips. Each of these tips looked like single tips with a large clean area and a heating neck. The sputtering process can therefore not be said to be entirely controlled. The ion-gun-sputtered and the self-sputtered tips exhibited the same characteristics, but we have chosen to use self-sputtering for two reasons. First, it is simpler in our experimental set-up, since we do not have to move the tip from the STM stage. Secondly, we can control the current and thereby detect decapitation.

Since scanning often changes the tip, we have found that a procedure of repeated heating–imaging–heating could help to maintain optimum imaging conditions. Migrating contaminants also seem to be removed by repeated heating. Finding the right heating procedure is a delicate matter. We preferred to heat the tip slowly and many times rather than just once quickly, in order to be certain not to accidentally melt the tip. However, it should be noted that the tip changes observable in STM imaging should not be observable with the TEM used in this investigation since they are on an atomic scale. When the tip was accidentally crashed during scanning, the tip was usually bent at a position where the tip has a diameter of about 170 nm (figure 12). From the shape it

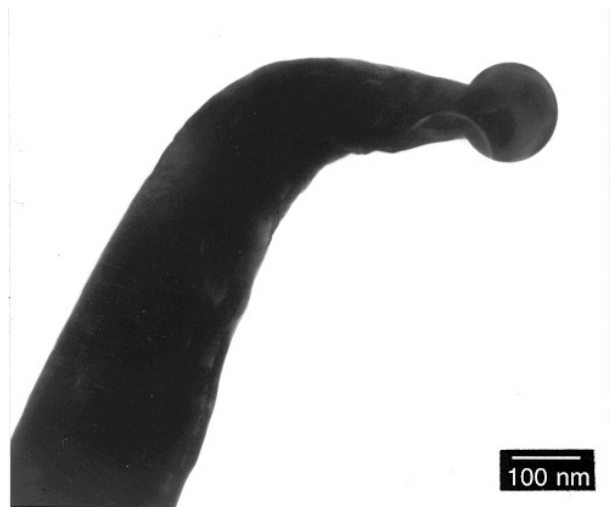


Figure 12. A tip that crashed during scanning. The tip was prepared by sputter decapitation and heating. The tip is bent where the diameter is about 170 nm and the bent part and the tip apex lie on a straight line, so that tunnelling possibly can occur at these two positions simultaneously.

seems plausible that tunnelling can occur from the bent part and the tip apex at the same time, thus causing highly unstable tunnelling conditions and blurred images which for us signify a crashed tip. Via decapitation it is possible to sharpen blunt tips by self-sputtering. However, we noted that one needs to have a high enough field-emission current in order to be able to sharpen the tip, at least when bringing the tip close to the counter electrode (filament). When the current was too low, sputter sharpening was very slow if it occurred at all.

The procedure of combined sputtering and heating is similar to the procedure suggested by Albrektsen *et al* [4]. They start by heating and continue with self-sputtering by Ne ions or sputtering by an Ar-ion gun at 0.5 keV. They show some TEM pictures of used tips having a thin oxide layer that they claim comes from storage and transport. We found that tips stored in air still were clean even down to the resolution of the TEM after a long storage time. A second TEM image of a sputtered tip that had been kept in air for more than 1 month before the second TEM imaging showed no visible traces of oxides. However, tips stored for several months showed some signs of oxidation. We can only speculate about why our observations are different from those of Albrektsen *et al*. It might be a question of resolution. Albrektsen *et al* note an oxide layer of less than 1 nm, which might be difficult for us to resolve in our TEM images. We also do not know how long the tips of Albrektsen *et al* had been stored. In our experience the oxidation in air is very slow, which means that UHV-cleaned W tips can not only be prepared in a separate vacuum chamber, but possibly also be used for STM in air.

After cleaning the image quality was improved; atomic resolution and low-noise spectra were regularly found. The absolute image quality is, however, dependent on the outermost atoms and cannot be predicted. The cleaning process does, however, dramatically raise the probability of having a favourable composition of the tip. However, a field ion microscope would give the atomic arrangement at the tip end and could possibly predict the quality of images.

5. Conclusions

Our investigations showed that STM imaging and spectroscopy can be improved by careful tip preparation. Judged from TEM images and field-emission data correlated to STM images, we found optimum W tips when our dc-etched tips are self-sputtered with Ne ions until decapitation or for a few seconds if no sharpening is required, or sputtered with 4 keV Ar ions for a few seconds. Sputtering is followed by heating to clean the very end and possibly rearrange the atoms at the tip apex. However, it is very important to be careful, since the result is highly dependent on the exact cleaning conditions and one has to find the proper procedure for the exact experimental configuration used. Checking field-emission currents gives one the ability to control the procedure and avoid melting. Field emission is also a reliable test of whether a tip has crashed. Chemical cleaning by HF solution produced tips that were unpredictably cleaned and ac polishing in neutol gave blunt and contaminated tips. The two latter methods were therefore not extensively investigated.

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