Tip Etching Kit User Manual

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Preface

This document has been compiled with great care and is believed to be correct at the date of print. The information in this document is subject to change without notice and does not represent a commitment on the part of Omicron NanoTechnology GmbH.

Notice

Some components described in this manual may be optional. The delivery volume depends on the ordered configuration.

Notice

This documentation is available in English only.

Notice

Please read the safety information on pages 8 to 9 before using the instrument.

Copyright

Warranty

Omicron acknowledges a warranty period of 12 months from the date of delivery (if not otherwise stated) on parts and labour, excluding consumables such as filaments, sensors, etc.

No liability or warranty claims shall be accepted for any damages resulting from non-observance of operational and safety instructions, natural wear of the components or unauthorised repair attempts.

Waste Electric and Electronic Equipment

In compliance with the WEEE directive (2002/96/EC) OMICRON ensures that all products supplied by OMICRON which are de-commissioned and which are labelled with a WEEE Registration Number will be taken back by OMICRON free of charge.

All costs of packing, transport, duty, etc. to the destination of the nearest OMICRON Returned-WEEE-Centre shall be borne by the customer. The customer is required to:

- Declare the returned material is free from contamination or hazardous materials from usage (include Decontamination Declaration sheet),
- Request a valid Return Material Authorisation (RMA) available from the OMICRON service department,
- Ship all returned goods to the advised destination "OMICRON Returned-WEEE-Centre, DDP (INCOTERMS)".

Otherwise OMICRON will not accept any shipment.

Normal Use

The **Tip Etching Kit** shall always be used

- with all cabling connected and secured, if applicable
- with all electronics equipment switched on
- in an indoor research laboratory environment
- by personnel qualified for operation of delicate scientific equipment
- in accordance with all related manuals.

Warning

Lethal Voltages!!

Adjustments and fault finding measurements as well as SPM experiments in environments other than UHV may only be carried out by authorised personnel qualified to handle lethal voltages.

Attention

Please read the safety information in the relevant manuals before using the instrument.

Conditions of CE Compliance

OMICRON instruments are designed for use in an indoor laboratory environment. For further specification of environmental requirements and proper use please refer to your quotation and the product related documentation (i.e. **all** manuals, see individual packing list).

The **OMICRON UHV System** complies with CE directives as stated in your individual delivery documentation if used unaltered and according to the guidelines in the relevant manuals.

Limits of CE Compliance

This compliance stays valid if repair work is performed according to the guidelines in the relevant manual and using original OMICRON spare parts and replacements.

This compliance also stays valid if original OMICRON upgrades or extensions are installed to original OMICRON systems following the attached installation guidelines.

Exceptions

OMICRON **cannot** guarantee compliance with CE directives for **components** in case of

- Changes to the instrument **not explicitly agreed by OMICRON**, e.g. modifications, add-on's, or the addition of circuit boards or interfaces to computers supplied by OMICRON.

The customer is responsible for CE compliance of entire **experimental setups** according to the relevant CE directives in case of

- Installation of OMICRON components to an on-site system or device (e.g. vacuum vessel),
- Installation of OMICRON supplied circuit boards to an on-site computer,
- Alterations and additions to the experimental setup not explicitly approved by OMICRON

even if performed by an OMICRON service representative.

Spare Parts

OMICRON spare parts, accessories and replacements are not CE labelled individually since they can only be used in conjunction with other pieces of equipment.

Notice

CE compliance for a combination of certified products can only be guaranteed with respect to the lowest level of certification. Example: when combining a CE-compliant instrument with a CE 96-compliant set of electronics, the combination can only be guaranteed CE 96 compliance.

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1. Introduction

The Tip Etching Kit is a device for producing sharp tungsten tips with smallest local radii of the tip apex. The so-etched tips are well suited for use in STM, FIM, FEM and SEM.

The tip etching kit is a hardware and electronics package consisting of the following components

- a control unit with adjustable etching voltage
- a set of cables
- an etching mount (tripod) with height adjustable rod
- three insets for tip reception
- a glass beaker
- a stainless steel electrode (= cathode) for DC etching in solution
- an Allen key 1.5 mm
- a pair of tweezers
- a pair of cutting pliers with hard metal inset
- tungsten wire (0.38 mm ∅)

The **tip etching device** shall always be used

- with all cabling connected and secured, if applicable
- with all electronics equipment switched on
- in an indoor research laboratory environment
- in accordance with all related manuals.
-

Attention
Please read the safety information in the relevant manuals before using the instrument.

2. Safety Information

Caution

Important:

- Please read this manual and the safety information in all related manuals before installing or using the instrument or electronics equipment.
- The safety notes and regulations given in this and related documentation have to be observed at all times.
- Check for correct mains voltage and grounding/earth before connecting any equipment.
- Do not cover any ventilation slits/holes so as to avoid overheating.
- The Tip Etching Kit may only be handled by authorised personnel.

Warning

Warning: Lethal Voltages!!

- Adjustments and fault finding measurements may only be carried out by authorised personnel qualified to handle lethal voltages.
- Lethal voltages are present inside the control unit.

Caution

Always

- All connectors which were originally supplied with fixing screws must always be used with their fixing screws attached and tightly secured.
- Always disconnect the mains supplies of all electrically connected units before
  - ⇨ opening a control unit case,
  - ⇨ touching any cable cores or open connectors,
- Leave for a few minutes after switching off for any stored energy to discharge.

Caution

NaOH Causes Severe Burns!

- In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
- Always wear suitable gloves and eye/face protection when handling NaOH solution.
- In case of accident or if feeling unwell remove victim to fresh air and seek medical advice immediately (show label when possible).
- Store and use in areas with adequate ventilation and away from acids.

Caution

This product is only to be used:

- indoors, in laboratories meeting the following requirements:
  - ⇨ altitude up to 2000 m,
  - ⇨ temperatures between 5°C / 41°F and 40°C / 104°F (specifications guaranteed between 20°C / 68°F and 25°C / 77°F)
  - ⇨ relative humidity less than 80% for temperatures up to 31°C / 88°F (decreasing linearly to 50% relative humidity at 40°C / 104°F)
  - ⇨ pollution degree 1 or better (according to IEC 664),
  - ⇨ overvoltage category II or better (according to IEC 664)
  - ⇨ mains supply voltage fluctuations not to exceed ±10% of the nominal voltage
- Condensation of humidity, particularly on water-cooled equipment, must be avoided.

3. General

STM tips are normally made from cut-to-size pieces of high purity metal wire. There are many different ways to make tips for atomic resolution, the very first STM tips simply were mechanically ground. A standard method for STM tip fabrication is electro-chemical etching.

Different tip materials and diameters require different etching methods while the tip shape can be influenced by varying the etching parameters. The metal wire for tip fabrication may be vacuum-annealed prior to etching, but polycrystalline tungsten wire as received from the manufacturer also yields atomic resolution tips. Tungsten tips may be etched in KOH or NaOH.

Notice

Tungsten wire pieces may be produced by cutting or breaking: Cut wire pieces perpendicular to the wire axis **prior to etching.** Attention: Tungsten wire is very hard and may damage your cutting device. Annealed tungsten wire can be fractured. **Do not cut the wire after etching!**

The supplied tungsten wire is as received from the manufacturer. You may want to anneal the tungsten wire prior to etching; to do so glow under vacuum (1×10^{-4} is sufficient) at a temperature of 2700 K for about 1 min.

After etching rinse the tips in distilled water and check for irregularities using an optical microscope. Additionally in-vacuum tip treatments, e.g. field emission, may be used if required.

The prepared STM tips are inserted into the tip carrier tube. Use the supplied pair of tweezers to fix the tip in the tube.

The following tip materials can be treated.

Tip Material	DC Solution	DC Lamella
polycryst. W	yes	yes
W(100)	yes	yes
W(111)	yes	yes
PtIr	no	no
Pt	no	no
Ir	no	no
Au	no	no

Table 1. Tip etching materials classified.

Advantages and Disadvantages of Etching Methods

The Tip Etching Kit is supplied with insets for holding the tip during etching. These are a bare wire inset for holding pieces of wire directly and two tip holders with adapters for different AFM and STM instruments.

In the tip holders a tungsten wire piece can be mounted prior to etching so that the complete tip holder may afterwards be introduced into the SPM without any further handling of the fragile tip. However, care must be taken not to wet the tip holder with the etching liquid or washing water, since considerable amounts of liquid may otherwise collect in the capillaries of the tip / tip holder assembly and spoil your vacuum afterwards.

This manual introduces two different etching methods for tungsten tips. The following table lists the advantages and disadvantages of these methods.

	DC Solution	DC Lamella
Advantage	• no supervision required • 80% of the tips achieve ideal shape	• ideal tip shape • smallest curvature • bottom end of tip may also be used
Disadvantage	• dissolving takes about 1 day at room temperature • meniscus may shift	• needs constant supervision

Table 2. Advantages and disadvantages of the etching methods.

The tip etching control unit in addition also offers two different current cut-off methods. These may be combined with the etching methods to give a total of three etching procedures described further on in the manual.

4. The Etching Control Unit

Front Panel

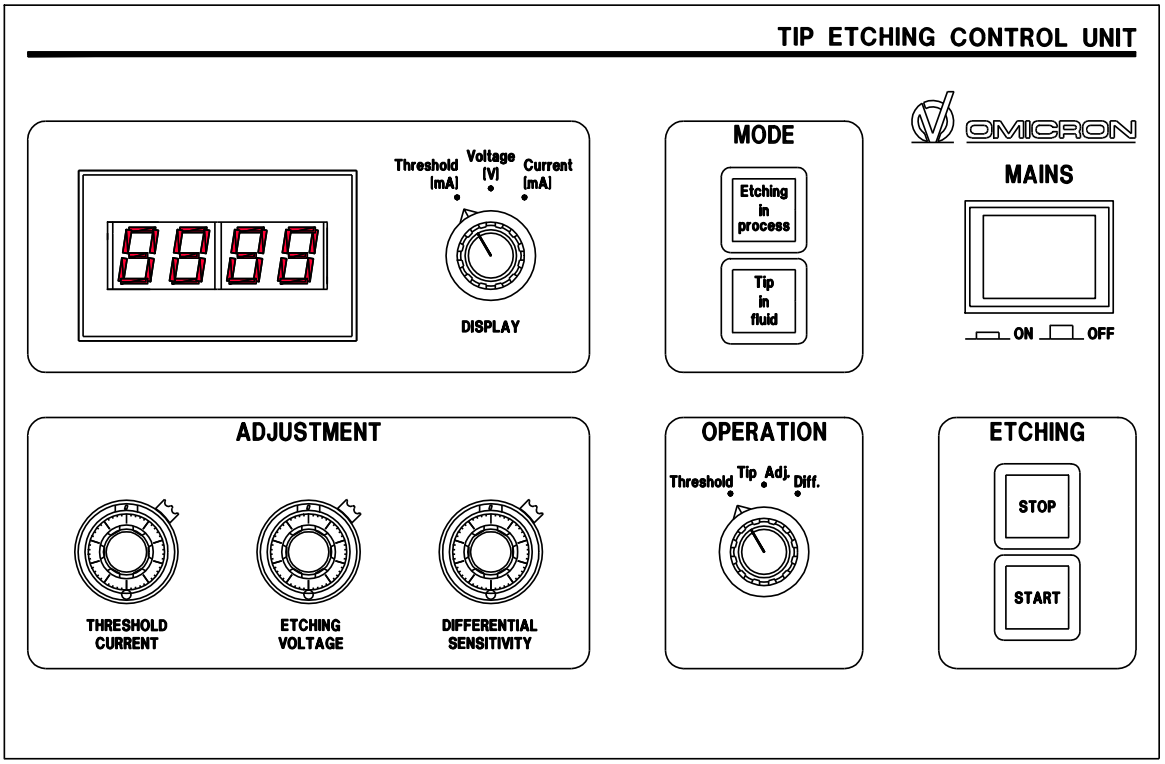
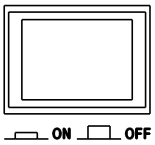
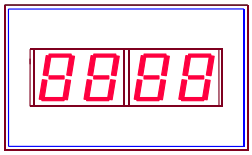
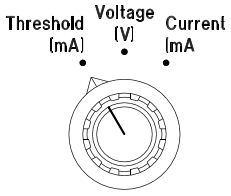
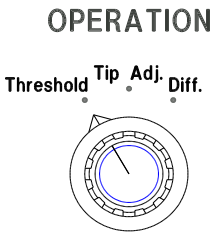
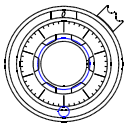
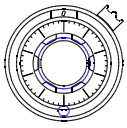
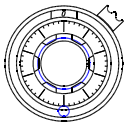


Figure 1. Control unit front panel, schematic diagram.

MAINS 	Push-button switch with optical status indicator.
	Green LED is lit during etching (DIFF. mode or THRESHOLD mode).
	Yellow LED is lit when wire touches the surface in TIP ADJ. mode.

	Selectable digital display for threshold current, etching voltage and etching current.
 DISPLAY	3-position rotary knob for selecting display signal to be threshold current in milliamps, etching voltage in volts, or etching current in milliamps.
	Push-button switch for starting the etching process.
	Push-button switch for stopping the etching process.
	3-position rotary knob for selecting etching with threshold function (responds to threshold current) tip adjustment for moving the tip into the liquid etching with differential limit stop (responds to current change)
 DIFFERENTIAL SENSITIVITY	Ten-turn potentiometer for pre-selecting the automatic switch-off sensitivity. This input is only active if the mode selector OPERATION is set to "Diff.". Range: 0 to 10 units.
 ETCHING VOLTAGE	Ten-turn potentiometer for setting the (nominal) voltage between tungsten wire (tip) and ring electrode continuously between zero and +10 V.
 THRESHOLD CURRENT	Ten-turn potentiometer for setting cut-off threshold between zero and 25 mA. This input is only active if the mode selector OPERATION is set to "Threshold".

Rear Panel

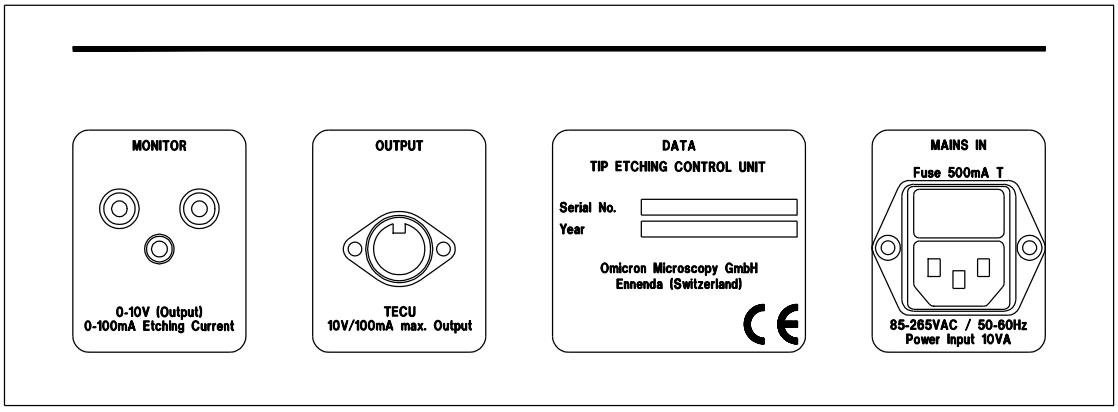
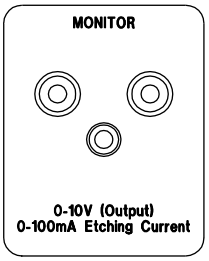
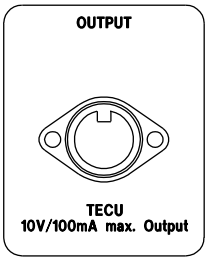
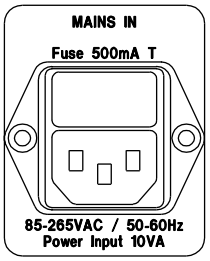


Figure 2. Control unit rear panel, schematic diagram.

	Banana monitor sockets for etching current: red = + black = - small = earth/ground 0-10 V output for 0-100 mA etching current maximum current output 20 mA
	3-pin Renk socket for connecting the control unit to the etching mount. 0-10 V output for 0-100 mA etching current. Resistive output impedance of the tip etching device is 110 Ω.
	**This unit is supplied with a 3-pin standard European mains socket** for AC input of 85-265 V ±10% and 50 or 60 Hz. Maximum power input 10 VA. The wire insulations of the standard 3-lead mains cable are coloured in accordance with the following code: Brown: Live Green/Yellow: Earth Blue: Neutral Mains fuse(s): 5 mm ∅, 20 mm long use 500 mA T for 85-265 VAC To change the fuse • disconnect mains • lift the flap using a screw driver for leverage • replace the fuse as indicated above • re-fit the fuse holder.

5. Cut-Off Procedures

In order to achieve high quality tips it is absolutely vital to cut the etching voltage in the instant the etched tip or excess end of wire drops down. The tip etching control unit offers two different methods for detecting the end of the etching process. Both methods employ the fact, that the etching current decreases when one end of the wire drops down.

Threshold Cut-Off

The threshold method sets a minimum current (threshold) and compares the etching current to this value. When the etching current is equal or below the threshold the etching voltage is switched off. This method is not very accurate as it cannot detect very small current changes. It is, however, rather simple to use and independent of small changes in NaOH concentration, wire length etc.

This method is well suited for DC etching in solution, it cannot be used for lamella etching (due to the small current changes involved)

Differential Cut-Off

The differential method sets a current variation ΔI (Diff.) and matches all current changes against this value. When the current changes by more than ΔI, the etching voltage is cut. This method can detect very small current changes. This fact may, however, also lead to premature cut-offs.

This method can be used for both etching recipes but is the only choice for the lamella etching procedure.

6. DC Etching in Solution (Threshold Cut-off)

Remarks concerning DC-etching of tunnel tips

- Prepare a NaOH-solution from 100 ml distilled or de-ionised water and 20 g NaOH pellets (see table 3). Attention: add pellets slowly! The pellets must be completely dissolved - at room temperature this takes about 24 hours.
- Fill the beaker of the tip etching device with part of the solution and insert the cathode into the etching liquid.
- Use 0.38 mm ∅ W-wire and cut (or break if annealed) off pieces for tunnel tips: final length + insertion depth (2 mm).
- Connect the electronics to the etching device using the provided cable set.
- Fix a piece of tungsten to a holder and insert this into the reception of the etching mount. Tighten the nuts firmly.
- Switch the control unit on and set the desired etching voltage.
- Switch the mode selector to "Tip Adjust" to move down the piece of tungsten into the centre of the ring electrode by turning the hand wheel.
- When the piece of wire touches the liquid surface (signal noise and "TIP IN FLUID" lights up) lower the tip by another 2 mm (about 2 turns on the hand wheel). Make sure the tip is adequately wet.

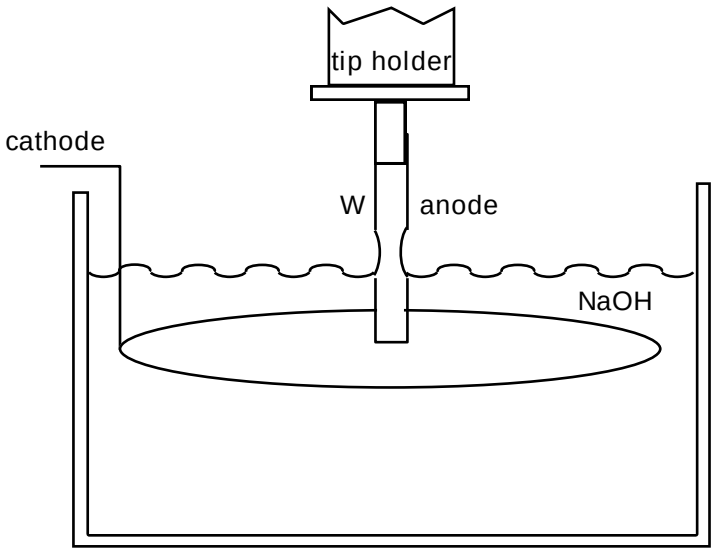


Figure 3. DC etching in solution, schematic diagram.

- Set the front panel display to show the threshold current and adjust the Threshold potentiometer for the desired stop current value, see table 3. If this current is reached the etching voltage is cut and the etching process stops. Upon automatic switch-off the electronics gives an optical and acoustic signal.
- Set the OPERATION selector to "Threshold" and press START to start the etching process.
- The etching process produces H_2 at the cathode and the tungsten wire is etched primarily at the liquid surface.
- Leave the tip in the solution until the end drops and the current ceases to flow. The power supply must be cut in the instant when the tip drops to ensure a sharp point.
- Remove the tip and dip it into distilled water at least 10 times.
- Check the shape of the tip with a microscope: for optimum quality, the tip should be a symmetric V-shape and the apex should not be resolved in an optical microscope at "× 500" magnification.
- Store tips in a dry environment (desiccator) or in vacuum.
- Thoroughly clean the tips with distilled water again directly before use, otherwise NaOH crystallites may remain on the point. Additionally in-vacuum tip treatments, e.g. field emission or vacuum annealing (several minutes at 1000°C and $p<10^{-4}$ mbar), may be used if required.

Table 3 gives an etching recipe.

Solution	20 g NaOH per 100 ml H_2O distilled
Ring electrode	stainless steel, diameter app. 36 mm, thickness 0.5 mm to 1.0 mm
U	3 V…10 V DC
I_{ini}	≈ 50 mA to 70 mA depending on insertion depth
I_{fin}	≈ 20 mA
Threshold	≈ 3 mA to 5 mA
Duration	5 min to 10 min depending on NaOH concentration, etching voltage and wire diameter

Table 3. Tip etching parameters for polycrystalline tungsten tips in solution.

Notice

For this etching procedure the differential cut-off method may also be employed

7. DC Etching in Solution (Differential Cut-off)

Remarks concerning DC-etching of tunnel tips

- Prepare a NaOH-solution from 100 ml distilled or de-ionised water and 20 g NaOH pellets (see table 3). Attention: add pellets slowly! The pellets must be completely dissolved - at room temperature this takes about 24 hours.
- Fill the beaker of the tip etching device with part of the solution and insert the cathode into the etching liquid.
- Use 0.38 mm ∅ W-wire and cut (or break if annealed) off pieces for tunnel tips: final length + insertion depth (2 mm).
- Connect the electronics to the etching device using the provided cable set.
- Fix a piece of tungsten to a holder and insert this into the reception of the etching mount. Tighten the nuts firmly.
- Switch the control unit on and set the desired etching voltage.
- Switch the mode selector to "Tip Adjust" to move down the piece of tungsten into the centre of the ring electrode by turning the hand wheel.
- When the piece of wire touches the liquid surface (signal noise and "TIP IN FLUID" lights up) lower the tip by another 2 mm (about 2 turns on the hand wheel). Make sure the tip is adequately wet.

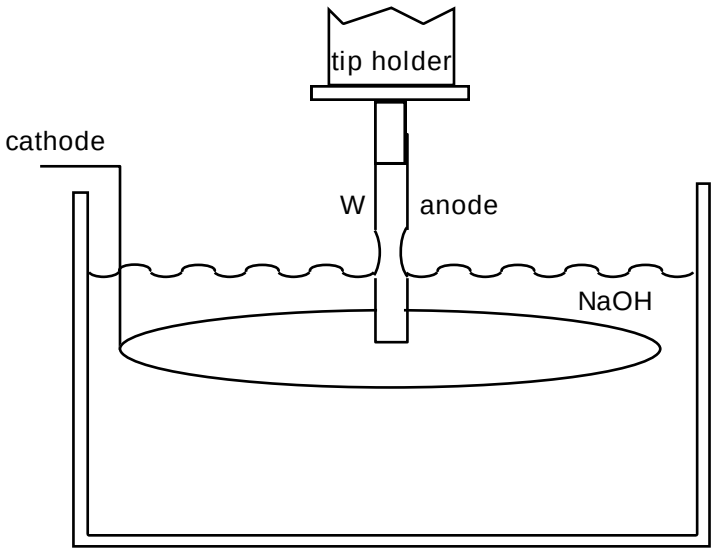


Figure 4. DC etching in solution, schematic diagram.

- Switch the mode selector to "Diff." and adjust the Sensitivity: potentiometer for the desired stop current difference ΔI, see table 4. If this value is reached the etching voltage is cut and the etching process stops. If the sensitivity is set too low the etching voltage may not be cut even when the tip drops. If the sensitivity value is set too high the etching process may be stopped even if the tip is not etched off completely. Upon automatic switch-off the electronics gives an optical and acoustical signal.
- Press START.
- The etching process produces H_2 at the cathode and the tungsten wire is etched primarily at the liquid surface.
- Leave the tip in the solution until the end drops and the current ceases to flow. The power supply must be cut in the instant when the tip drops to ensure a sharp point.
- Remove the tip and dip it into distilled water at least 10 times.
- Check the shape of the tip with a microscope: for optimum quality, the tip should be a symmetric V-shape and the apex should not be resolved in an optical microscope at "× 500" magnification.
- Store tips in a dry environment (desiccator) or in vacuum.
- Thoroughly clean the tips with distilled water again directly before use, otherwise NaOH crystallites may remain on the point. Additionally in-vacuum tip treatments, e.g. field emission or vacuum annealing (several minutes at 1000°C and $p<10^{-4}$ mbar), may be used if required.

Table 3 gives an etching recipe.

Solution	20 g NaOH per 100 ml H_2O distilled
Ring electrode	stainless steel, diameter app. 36 mm, thickness 0.5 mm to 1.0 mm
U	3 V…10 V DC
I_{ini}	≈ 50 mA to 70 mA depending on insertion depth
I_{fin}	≈ 20 mA
Sensitivity	≈ 6.0
Duration	5 min to 10 min depending on NaOH concentration, etching voltage and wire diameter

Table 4. Tip etching parameters for polycrystalline tungsten tips in solution.

Notice

For this etching procedure the threshold cut-off method may also be employed.

8. DC Etching "Lamella"

Notice

The ring electrode required for lamella etching, see figure 5, is not supplied with the instrument.

- Prepare a NaOH-solution from 100 ml distilled or de-ionised water and 8 g NaOH pellets (see table 5). Attention: add pellets slowly! The pellets must be completely dissolved - at room temperature this takes about 24 hours.
- Fill the beaker of the tip etching device with part of the solution and insert the cathode into the etching liquid.
- Use 0.38 mm ∅ W-wire and cut (or break if using annealed wire) off pieces for tunnel tips: final length + insertion depth (≈ 5 mm).
- Or use pieces with 2 × final length for etching two tips in one go. In this case catch the bottom tip in a thick foam, e.g. shaving lather.
- Connect the electronics to the etching device using the provided cable set.
- Switch the control unit on and set the desired etching voltage.
- Dip the electrode into the solution by lifting the filled glass beaker.
- Move down the piece of tungsten into the centre of the ring electrode. The tip should extent about 5 mm through the ring.

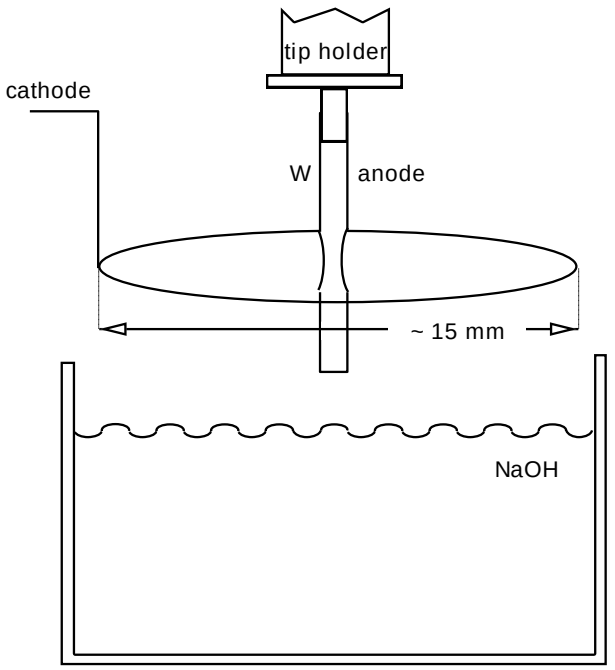


Figure 5. DC etching in lamella, schematic diagram.

- Adjust the Sensitivity: potentiometer for the desired stop current difference ΔI, see table 5. If this value is reached the etching voltage is cut and the etching process stops. If the sensitivity is set too low the etching voltage may not be cut even when the tip drops. If the sensitivity value is set too high the etching process may be stopped even if the tip is not etched off completely. Upon automatic switch-off the electronics gives an optical and acoustical signal.
- Switch the mode selector to "Diff." and press START.
- The lamella breaks rather often during etching (i.e. three to five times for one tip) in this case wet the ring to restore the lamella and proceed.
- Leave the tip in the solution until the end drops and the current ceases to flow.
- Remove the tip and dip it into distilled water at least 10 times.
- Check the shape of the tip with a microscope: for optimum quality, the tip should be a symmetric V-shape and the apex should not be resolved in an optical microscope at "$\times$ 500" magnification.
- All vibration of the etching device should be avoided during etching.
- At the end of the etching process the end of the tungsten piece will drop. The power supply must be cut in this instant to ensure a sharp point.
- Store tips in a dry environment (desiccator) or in vacuum.
- Thoroughly clean the tips with distilled water again directly before use, otherwise NaOH crystallites may remain on the point. Additionally in-vacuum tip treatments, e.g. field emission or vacuum annealing (several minutes at 1000°C and $p<10^{-4}$ mbar), may be used if required.

Solution	8 g NaOH per 100 ml H_2O distilled
Ring electrode	gold ring, diameter app. 15 mm, thickness ≈0.5 mm
U	≈ 4 V DC
I_{ini}	≈ 20 mA
I_{fin}	≈ 2 mA
Sensitivity	≈ 6.0

Table 5. Tip etching parameters for polycrystalline tungsten tips in lamella.

9. Trouble Shooting

Etching Process Takes Longer Than 15 Minutes

- The NaOH has not yet completely dissolved. In this case wait until completely dissolved before starting the etching process.
- The NaOH concentration is not uniform over the entire volume. In this case stir the solution and start again.

DC Solution: Concave Meniscus Slides Down

With time the solution level may decrease and lead to the formation of a second meniscus, see figure 6. However, tips as shown in situation ③ may still be used for STM imaging.

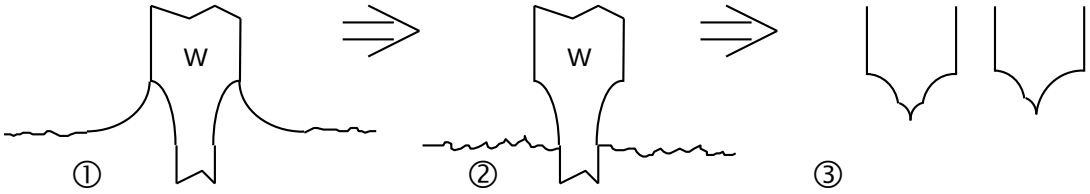


Figure 6. Meniscus slides down.

To prevent the formation of a second, lower meniscus try one of the following:

- Add NaOH solution to reconstitute situation ①.
- Adjust the Z-position of the tip to reconstitute situation ①.
- Increase the NaOH concentration in order to accelerate the etching process.

DC Lamella: Lamella Breaks

This happens rather often during lamella etching (i.e. three to five times for one tip), requiring constant supervision of the etching process.

- Avoid all contamination of the etching solution in order to have a high surface tension.
- Wet the ring to restore the lamella and proceed.

10. Literature

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Service at Scienta Omicron

Should your equipment **require service**

- Please **contact Scienta Omicron** headquarters or your local Scienta Omicron representative to discuss the problem. An up-to-date address list is available on our website

  http://www.scientaomicron.com/

- Make sure all necessary information is supplied. Always **note the serial number(s)** of your instrument and related equipment (e.g. head, electronics, preamp…) or have it at hand when calling.

If you have to **send any equipment back to Scienta Omicron**

- Please contact **Scienta Omicron headquarters** before shipping any equipment.
- Place the instrument in a polythene bag.
- **Reuse the original packaging and transport locks.**
- Take out a **transport insurance policy.**

For ALL vacuum equipment:

- Include a filled-in and signed copy of the "Declaration of Decontamination" form which can be found at the back of the equipment manual.

No repair of vacuum equipment without a legally binding signed decontamination declaration !

- Wear suitable cotton or polythene gloves when handling the equipment.
- **Re-insert all transport locks (**if applicable**).**
- Cover the instrument with aluminium foil and/or place it in a polythene bag. Make sure no dust or packaging materials can contaminate the instrument
- Make sure the **plastic transport cylinder (**if applicable**) is clean**.
- Fix the instrument to its plastic cylinder (if applicable).

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