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Tip apex shaping of gas field ion sources

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ABSTRACT

A procedure to control W(111) tip shape during etching to a single atom is described. It is demonstrated that the base of a single atom tip (SAT) can be shaped in order to alter the final operating voltage and emission opening angle of single atom tips for use as gas field ion sources or electron cold field emission sources. The operating voltages for single atom tips varied between 5 and 17 kV during helium ion beam generation. The emission properties of SATs were evaluated by fitting SAT images and measuring the full width at half maximum (FWHM) of the helium ion images. The FWHM is related to the linear opening angle and was evaluated as a function of SAT operating voltage. The results show that a forward focussing effect is observed such that the spot size decreases faster than is expected solely from an acceleration effect, indicating an affect from the tip shape. These results have consequences in designing gas field ion sources where etching is used to prepare the emitter.

1. Introduction

There has been significant interest in developing suitable sources for scanning ion microscopy (SIM). These ion sources must be easy to fabricate and readily rebuilt without removal from the microscope. Ideally, each rebuild must result in a functionally identical apex structure such that emission properties, including the emission axis, are unchanged, thereby eliminating the need for substantial gun alignment. The source must support large ion currents and be stable for extended periods of time. In order to improve performance of the microscope, it is also desirable that angular current intensity of the ion beam be as large as possible in order to maximize probe current. One route to improving this is by reducing the beam opening angle by the preparation of a very small protrusion on a large radius tip. The large base is thought to forward focus the beam and decrease the linear beam opening angle [1–6]. By decreasing the opening angle, there is potential to increase the angular current intensity. Optimizing the shape of the tip of gas field ion emitters has also been discussed in the context of creating larger ion currents [7,8].

As described previously, the nitrogen assisted etching process enabled the formation of single atom tips (SATs) [9,10]. In that previous work, the global shape of the tip was determined solely by the initial radius of the tip. No additional control over the etching mechanism was suggested and the tip shape was determined by continuous etching as tip voltage was slowly lowered. During etching the voltage is controlled such that the apex atoms are not evaporated, creating a high aspect ratio tip with a lower applied voltage for the SAT, ~4–6 kV. Some information about the structure of these tips has recently been described using neon as an imaging gas to help elucidate the tip structure of W(111) nanotips during the etching process [11]. Oxygen has also been shown to be a viable etchant gas for the creation of nanotips and a constant voltage etching method has been described for the creation of nanoprotrusions [12–14]. Sugiura et al. also discussed the helium beam opening angle from trimer tips but did not compare SATs or tips of varying base diameters [14]. Water has also been found to aggressively etch tungsten tips and has been used with a constant voltage etching method, along with conventional etching, for the preparation of tips for field emission studies [15]. However, SATs were not prepared using field assisted etching methods and the ion emission characteristics of the various tips were not discussed.

In this manuscript we build upon our original etching process by controlling and tailoring the voltage ramp rate and nitrogen pressure in order to create customized tip shapes. This procedure allows for the shaping of the supporting tip base of the SAT during the etching procedure. The method can be applied to create SATs with various operating voltages, by creating a range of high and low aspect ratio tips, based on the etching parameters. The ability to shape the supporting base of the emitter can also lead to the ability to tailor emitters with different lensing effects which is important for both ion and electron emission. For example, a divergent beam is desired for point projection holography [16] while confined beams are desired in SIM applications.

We have prepared SATs with final imaging voltages between 5 kV and 17 kV for this study. The full range of possible operating

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voltages has not yet been explored. We also assessed the effect of the base tip shape on the forward focussing effect of nanotips prepared by the nitrogen assisted field evaporation etching method [9–11]. Tips of varying diameter were prepared by the controlled etching procedures and the SAT images were measured and compared. We show that there is a tip base forward focusing effect on the helium ion linear opening angle from SATs leading to the potential for improved angular current intensity.

2. Experimental setup

Experiments were carried out in an ultrahigh vacuum system with base pressure $<1\times10^{-10}$ Torr. Tips were cooled to liquid nitrogen temperature using a flow-through cryostat utilizing cold nitrogen gas. All voltages were controlled and monitored by a computer. The field ion microscope (FIM) pattern was amplified by a Hamamatsu microchannel plate (MCP) and displayed on a phosphor screen. Images were recorded using a PixelFly high sensitivity camera. SAT images were fit to 2-D Gaussian profiles and the FWHM was determined from the fit.

Electrochemically etched W(111) tips were supported by a wire loop for heating and installed in the FIM chamber. Tips were degassed at 900 °C for 3 min, cooled and field evaporated to prepare the initial tip. Nitrogen gas pressure for etching was controlled with a variable leak valve to uncorrected pressures between 1×10^{-8} and 5×10^{-6} Torr.

3. Results and discussion

3.1. Dynamics of nitrogen etching and tip shape control

The nitrogen etching of the tungsten surface occurs at an applied tip field below a critical value, V_{crit}, and above a threshold voltage, V_{min}. In case of helium as an imaging gas

$$V_{\text{fe}} > V_{\text{img}} > V_{\text{crit}} > V_{\text{min}}$$

where V_{img} is the best imaging voltage and V_{fe} is the voltage where field evaporation takes place. V_{crit} is related to the ionization probability of nitrogen molecule while V_{min} corresponds to the lowest electric field to remove (field evaporate) “etched” tungsten atoms. At the start of etching, a tip field (voltage) close to the best imaging voltage is generally selected. This defines an “etching band” where applied voltage V satisfies $V_{\text{crit}} > V > V_{\text{min}}$. As etching progresses the tip apex becomes protruded and local electric field increases. At this point, the etching program can take on varying modes, including *constant field* or *forced evaporation* modes.

In the *constant field* method, the tip voltage is lowered to prevent field evaporation of apex atoms ($V_{\text{fe}} > V$) and the continued lowering of V allows the last atoms to be maintained. As etching continues at the shank the tip apex becomes narrow, eventually leaving a single tungsten atom at the apex. This results in a high aspect ratio nanotip on a base tip radius that is largely unaltered. A tip prepared with the *constant field* method is shown in Fig. 1a.

In the *forced evaporation* method, once the initial nitrogen etching is established, the tip voltage is lowered slowly, apex atoms can field evaporate as the tip sharpens. This *forced evaporation* happens because V approaches V_{fe} at the apex as the tip etches. The evaporation leads to a flattening of the overall tip shape. Continued etching and field evaporation will eventually lead to a nanotip with a relatively low aspect ratio and hence high tip operating voltage. A tip prepared using the *forced evaporation* method is shown in Fig. 1b.

It is also possible to fine tune the etching process such that a combination of both the *constant field* and *forced evaporation* methods are used simultaneously to control the overall shape of the tip. This is achieved by modifying the rate at which the voltage is changed during the etching process. If the voltage is dropped more quickly, nitrogen etching is faster than apex field evaporation and a taller nanotip is formed. If the voltage is dropped more slowly, paused or adjusted through a custom ramping profile, shank and apex atoms are controllably removed and the tip shape will be controlled through a balance of etching and field evaporation. The nitrogen pressure may also be adjusted to vary the etching rate. These controls help to define the shape of the tip and the final operating voltage of the SAT.

Fig. 1 shows two single atom tips prepared by the above methods as well as a schematic of the tip shaping. Both tips started with a measured radius of ~12 nm (estimated from a number of net rings between [111] and [211] directions) and are etched to single atoms. However, the overall tip shapes are substantially different as are the final voltages, found to be 8 kV and 13 kV, respectively The 8 kV single atom tip was prepared by attempting to maintain the apex atom during the etching (faster voltage drop) while the 13 kV single atom tip was prepared by a slow voltage drop which allowed field evaporation during the etching process. Although the operating voltages are quite different for each single atom tip, the electric field at the apex atom is similar, as known by the helium ionization.

3.2. Spot size analysis

It is also evident that the spot size is quite different for the two single atom tips shown in Fig. 1 with the higher operating voltage tip producing a smaller spot. The question now arises: what are the contributing factors to the spot size differences? Is it a result solely of the larger acceleration voltage or is there an effect of the emitter base size or shape?

In order to evaluate the tips, we have compared multiple SATs at various operating voltages. The intensity profile of the spot was to fit a 2D Gaussian (Eq. (1)) and the full width at half maximum (FWHM) was measured from the fit. In all cases the σ_x and σ_y were set to be equivalent in order to average slight asymmetries in the data set. The FWHM is proportional to σ: $\text{FWHM}=2(2\ln 2)^{0.5}\sigma$. The FWHM is directly proportional to the linear opening angle of the helium beam given a known geometry between the tip and the imaging screen.

$$Z=Z_0+A\exp\left(-\left(\frac{(x-x_0)^2}{2\sigma_x^2}+\frac{(y-y_0)^2}{2\sigma_y^2}\right)\right) \tag{1}$$

Fig. 2 shows an example of one of the SAT image fittings. Fig. 2a shows the helium ion image from as SAT operating at 17 kV and Fig. 2b shows the Gaussian fit with the experimental data shown in color and the fit shown in the wire mesh.

Multiple SATs were then prepared using a combination of the *constant field* and *forced evaporation* methods described above. Two sets of data were collected in two independent FIM systems with different geometric setups; however, geometries were unchanged within a single instrument. This provided data sets of SATs operating at various voltages. A plot of the FWHM of SATs as a function of beam voltage is shown in Fig. 3. The figure shows a relationship where the FWHM decreases for tips prepared at higher acceleration voltages. Fits for the data sets from the two instruments were performed. A fit of the data from instrument 1 shows a relation of $V^{-0.7\pm0.1}$ while the second instrument shows a relation of $V^{-0.62\pm0.02}$. Instrument 1 prepared a larger number of tips over a smaller voltage range. Some scatter in the data may be attributed to the difficulty in determining a best imaging voltage