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Fabrication of a trimer/single atom tip for gas field ion sources by means of field evaporation without tip heating

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ABSTRACT

A gas field ion source (GFIS) has many advantages that are suitable for ion microscope sources, such as high brightness and a small virtual source size, among others. In order to apply a tip-based GFIS to an ion microscope, it is better to create a trimer/single atom tip (TSAT), where the ion beam must be generated in several atoms of the tip apex. Here, unlike the conventional method which uses tip heating or a reactive gas, we show that the tip surface can be cleaned using only the field evaporation phenomenon and that the TSAT can also be fabricated using an insulating layer containing tungsten oxide, which remains after electrochemical etching. Using this method, we could get TSAT over 90% of yield.
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1. Introduction

The application of the helium ion microscope (HIM) has expanded in various fields, including nano-patterning and material science and biology, due to the high spatial resolution of these devices for imaging and their compatibility with high-precision machining [1–4]. The HIM uses GFIS, and the ion beam is generated by the ionization of gas molecules near the tip apex. The GFIS has a much smaller virtual source size than a liquid metal ion source (LMIS). The energy spread of the GFIS is predicted to be less than 1 eV, and this estimated value indicates that it offers high brightness and a high current density source [5,6]. Furthermore, the probe beam size can be made much smaller than that of LMIS, allowing sub-nanometer imaging and the fabrication of sub-10 nm structures. In addition, it can minimize the chemical interaction with the sample through the use of noble gases such as He, Ne, and Xe. However, it is difficult to create atomically sharp tips to implement this high-performance GFIS, as ionization phenomena must be induced in only a few atoms to obtain high beam current density that is involved in the quality of the imaging.

TSAT can be produced by either a field-assisted reactive gas etching method based on oxygen or nitrogen or a build-up method [7–10]. However, these methods usually require a tip cleaning process at an approximate temperature of 1000 K before the TSAT creation process. During this heating process, the tip is usually welded to a heating loop and heated by a resistive heating method. Therefore, the tip holder has to be configured such that it allows current to flow. In order to apply a GFIS to an ion microscope, an ultra-high vacuum (UHV) environment is required for stable beam generation and to prevent the tip from becoming contaminated. Also necessary is high voltage of several tens of kV to accelerate the beam, as well as tip cooling for a high beam current. Thus, it is better to simplify the GFIS configuration when attempting to ensure these conditions.

In this study, we show that TSAT can be created via the field evaporation effect with an oxide layer which remains on the tip surface owing to the absence of a tip-cleaning process. The proposed method has fewer steps than the conventional method and does not heat the tip, simplifying the structure of the experimental apparatus as compared to that of the conventional method.

2. Experimental setup

We fabricate an ultra-sharp tip with a curvature of several to several tens of nanometers, and the tip is further etched to produce a TSAT in a vacuum condition. Ultra-sharp tips are usually

Abbreviations: GFIS, Gas field ion source(s); TSAT, Trimer/single atom tip; FIM, Field ion microscope; BIF, Best image field.

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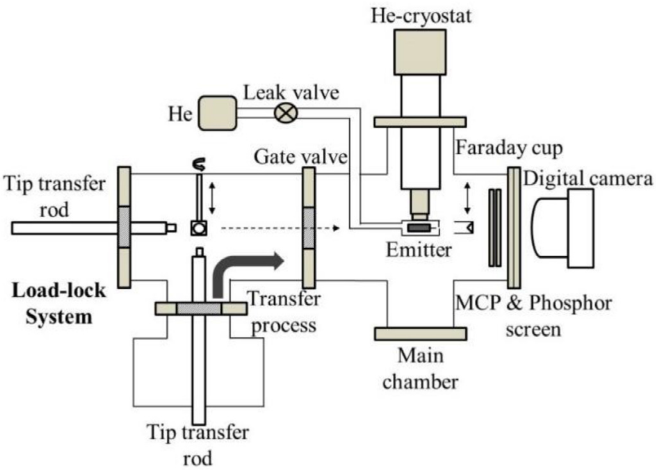

Fig. 1. Schematic diagram of the experimental system. A tip was transferred by a transfer rod from the first chamber to the main chamber. The ion beam patterns are imaged with a combination of MCP, a phosphor screen and a digital camera. The beam current can also be measured using a Faraday cup positioned between the emitter and the screen. A helium cryostat was installed to lower the tip temperature.

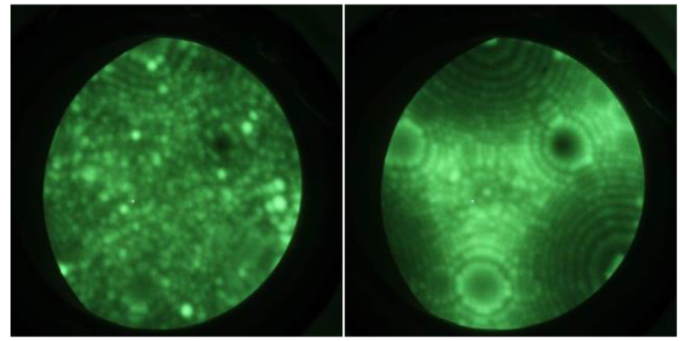

Fig. 2. FIM images of the W(111) tip. (a) Before applying a high electric field, the surface of the tip is covered with insulation layers. (b) After the field evaporation process, the tip surface becomes clean and the crystalline structure appears.

manufactured by electrochemical etching method using tungsten wire of which diameter is several hundreds of µm. In order to observe the state and changes of the surface of the tip during the TSAT fabrication process by a field ion microscope (FIM), the experimental setup shown in Fig. 1 was utilized. Implementing a UHV in a single chamber requires a baking process for every change of the tip because the vacuum chamber is exposed to ambient air. Thus, a load-lock system was constructed using three chambers to exchange tips without a time-consuming baking process, and the tips were transferred in a stepwise manner through a transfer rod to a tip holder in the main chamber. The ultimate pressures of the three chambers are $\sim 10^{-7}$ mbar, $\sim 10^{-9}$ mbar, $\sim 10^{-10}$ mbar. Furthermore, the system consists of a differential pumping system to introduce gas at a pressure higher around the tip than in the main chamber, as the generated ion beam current is linear with the injected gas pressure. We created a tip with a radius of curvature of approximately 20 nm from single crystal W(111) wire and polycrystalline tungsten wire of which the diameters were 250 µm using an electrochemical etching method. Potassium hydroxide (KOH, 4.5 M) was used as the electrolyte. The etched tip was fixed in the tip holder in the main chamber, and the FIM pattern was observed by a micro-channel plate (MCP) and a digital camera (Cannon EOS-600D). He gas was injected into the chamber until the chamber pressure reached 4.0×10^{-8} mbar. When the temperature of the tip is reduced, the generated ion beam increases owing to the increased ionization rate near the tip apex. Hence, a cryostat system (Sumitomo CH-204) was used to cool the tip to a temperature of close to 34 K. A movable Faraday cup was installed between the tip and the MCP to measure the ion beam current.

3. Results and discussion

3.1. Tip cleaning and TSAT making

In general, the tungsten tip surface can be cleaned not only by tip heating but also by field evaporation [11–13]. We adopted only the field evaporation process with which to clean the tip surface. Fig. 2 shows the cleaning process of the tip surface coated with an insulating layer by means of electrochemical etching and exposure to the ambient air, which was conducted in a vacuum with the field evaporation phenomenon and without a high-temperature tip heating process [14,15]. With a 12 kV extraction voltage, we observed the field evaporation cleaning process and maintained the applied voltage for ten minutes. As shown in Fig. 2(b), the crystal structure of tungsten is clearly visible. The left part of the FIM image in Fig. 2 appears to be shrouded because the ion beam path is screened by the radiation shield of the cryostat located in front of the MCP.

The best image field (BIF) is the image in which the FIM is most clearly visible. When we maintained the voltage of the tip when observing the FIM pattern under the BIF condition, we found the ‘etch-like’ phenomenon near the tip apex, and the FIM image became smaller. To the best of our knowledge, the BIF condition is generally lower than the evaporation field. Thus, no etching occurs. Accordingly, we observed the FIM in real time while lowering the voltage gradually, but the etch-like phenomenon was still observed. As shown in Fig. 3, the TSAT was fabricated by adjusting the applied voltage accurately so that no abrupt changes would occur. At this time, the temperature of the tip was approximately 83 K. Because of the generation of the ion beam increases in intensity as the tip temperature is lowered, the temperature was set to 83 K and the FIM image was monitored. The tip apex area became smaller due to field evaporation as time elapsed. We confirmed that the tip became sharper as the FIM image became smaller, but we used the ring count method to measure the radius of curvature of the etched tip more accurately. The ring count method refers to a means of estimating the radius of curvature of the tip by counting the number of rings between two specific crystal orientations in the FIM image [16–18]. Using this method, we could estimate the radius of curvature of Fig. 3(a) and (b). The corresponding calculated values were 13.26 nm and 6.03 nm. In the case shown in Fig. 3(c)–(f), it was confirmed that less than 20 atoms remain on the tip surface in the FIM image; hence, we did not undertake a calculation with the ring count method. We confirmed that the decreasing FIM image area indicates that the tip is being sharpened. As the tip is sharpened by this phenomenon, the field enhancement effect increases and the field evaporation phenomenon then occurs much more rapidly. To etch the tip stably, we controlled the speed of the etch-like phenomenon by changing the applied voltage of the tip in full detail when we observed only 20–40 atoms on the tip surface. As shown in Fig. 3(d), we observed the trimer tip in the 9 kV condition. Moreover, as indicated in Fig. 3(e), the single-atom tip appeared at 8 kV. If the topmost atom of SAT was evaporated, we observed nothing except a dark area. If we increase the voltage slightly, we can make the trimer tip again, as shown in Fig. 3(f). After the SAT was destroyed, we regenerated the SAT five times repeatedly through a field evaporation process. However, we

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