

Novel Scanning Ion Microscope with H₃⁺ Gas Field Ionization Source

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It has been well described that three kinds of hydrogen ion species (H⁺, H₂⁺, and H₃⁺) can be contained in an ion beam emitted from a hydrogen Gas Field Ionization Source (GFIS) [1], and the protonated hydrogen ion (H₃⁺) has been considered as a minor constituent. However, we have surprisingly discovered that the H₃⁺ emission can be dominant in a special condition mentioned below.

A scanning ion microscope (SIM) equipped with the GFIS with a very high brightness and a small sized (~0.3 nm) source has been commercialized [2]. Variety of practical usages and valuable studies using the microscope has been reported so far [3]. The GFIS can emit different ion species by replacing its ionizing gas. The commercialized GFIS-SIM can select the helium ion for visualization of surface of specimen with a sub-nm resolution. It can also select the neon ion for higher precision nano-machining than Ga-FIB, which takes advantage of higher sputtering yield due to larger mass of ions. Similarly, we have developed a home-made SIM machine with a hydrogen GFIS for the purpose of suppressing a damage during observation comparing with the helium ion.

During SIM observing of Si patterns on a SiO₂ substrate, we have found that the sample image splits into triplet possibly by a stray magnetic field (Fig. 1(a) and (b)). It indicates that the ion beam contains three kinds of ion species with different mass to charge ratios, which would be deflected by three different angles. These deviations of ion beams would result in triple separate SIM images. We have plotted the acceleration voltage dependence of separation length between edges and verified that these separation lengths are inversely proportional to square root of acceleration voltages (Fig. 1(c)). This result agrees with our speculation that the magnetic deflection effect dominantly causes these separations. We have also estimated that the magnetic field to make these deflections (~100 nm) is comparable to the geomagnetic field or the magnetic field by an ion pump. Therefore, we can successfully identify the ion of these three (H⁺, H₂⁺, and H₃⁺) to generate each of three images based on the mass to charge ratios. From our SIM images, we have further found that the H₃⁺ can be the dominant constituent of the ion beam under a certain condition contrary to past results obtained by a pulsed-laser field desorption measurement [1].

We investigated how the amount of H₃⁺ can be enhanced. First, we checked the atomic structure of the emitter tip apex because the past experiments reported that the atomic protruding on the tip surface was an important factor of H₃⁺ detection [1]. We fabricated atomic structures at the W<111> oriented tip apex by field evaporation, and varied the number of atoms on the tip apex. From the extraction voltage dependence of the probe current shown in Fig. 2(a), there are two pronounced peaks under the condition where the emitter tip apex is terminated by a single atom with no adjacent atoms (red curve in Fig. 2(a)). At the lower extraction voltages below the first peak (<9 kV), H₂⁺ emission is dominant. The amount of H⁺ ions increases at the voltage above the first peak (>10 kV). This transition of ion species is expected from the past report [1]. But within the extraction voltage for the second sharp peak, we discovered that the amount of H₃⁺ was remarkably enhanced contrary to expectation. We have been able to take SIM images on this condition (Fig. 2(b)). In this picture there was almost no sign of H⁺ or H₂⁺.

On the other hand, we could not detect the second peak in the case of many-atom termination. The black curve in Fig. 2(a) shows extraction voltage dependence of the probe current in the case of eight-atom tip. Two- or three-atom tip can emit H_3^+ but the amount is smaller than the case of the single atom termination. In our measurement, the probe current is always restricted within the same solid angle and to the same direction respect to the emitter tip. In our field ion microscope images, the direction of H_3^+ emission from three-atom tip slightly deviates from the direction of H_2^+ . This shift of emission direction can cause the decrease of H_3^+ current. We think structure dependence of H_3^+ emission is the key to understand how this unusual ion is generated, and further studies are necessary.

References:

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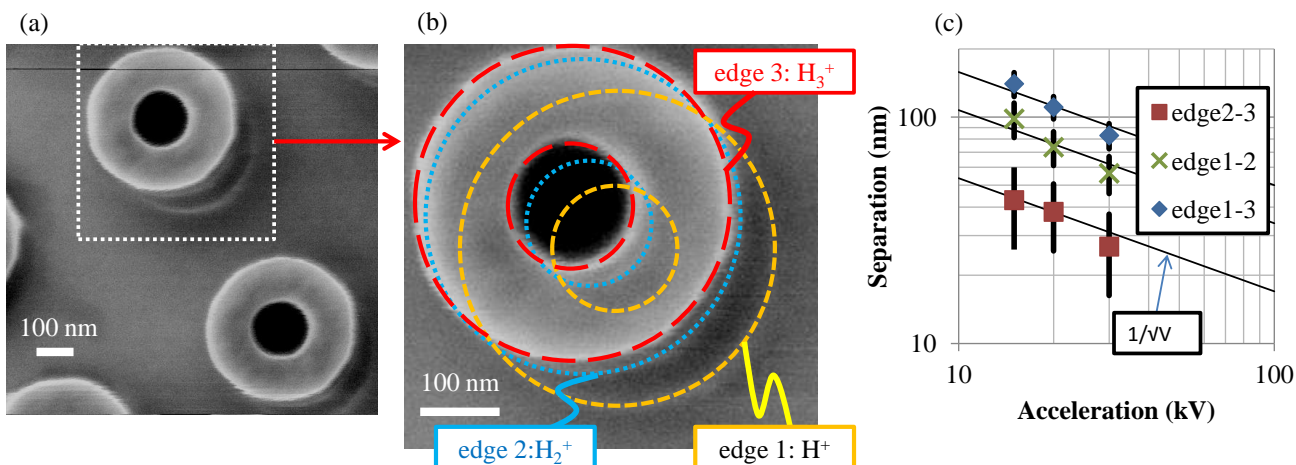


Figure 1. (a) AnSIM image obtained by a hydrogen ion beam. Individual edges are highlighted by color circles in (b). (c) Acceleration voltage dependence of separations between each edge images.

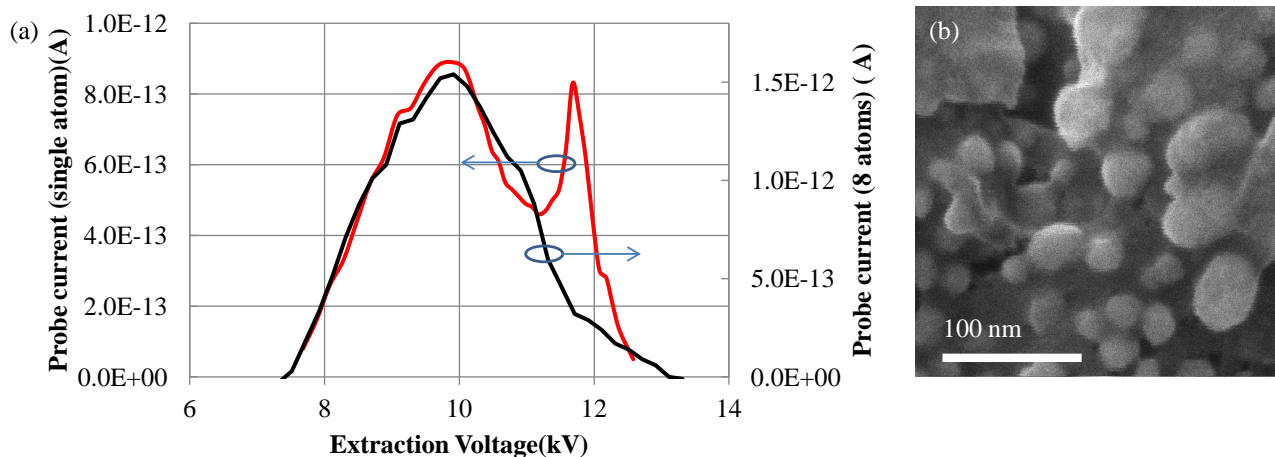


Figure 2. (a) Extraction voltage dependence of the probe current. A pronounced second peak emerges in the case of single-atom termination (red line), whereas only one peak was observed for the 8-atom tip. (b) An H_3^+ SIM image of Au nano-particles.