Fabrication of electron beam deposited tip for atomic-scale atomic force microscopy in liquid

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Abstract
Recently, possibilities of improving operation speed and force sensitivity in atomic-scale atomic force microscopy (AFM) in liquid using a small cantilever with an electron beam deposited (EBD) tip have been intensively explored. However, the structure and properties of an EBD tip suitable for such an application have not been well-understood and hence its fabrication process has not been established. In this study, we perform atomic-scale AFM measurements with a small cantilever and clarify two major problems: contaminations from a cantilever and tip surface, and insufficient mechanical strength of an EBD tip having a high aspect ratio. To solve these problems, here we propose a fabrication process of an EBD tip, where we attach a 2 μm silica bead at the cantilever end and fabricate a 500–700 nm EBD tip on the bead. The bead height ensures sufficient cantilever-sample distance and enables to suppress long-range interaction between them even with a short EBD tip having high mechanical strength. After the tip fabrication, we coat the whole cantilever and tip surface with Si (30 nm) to prevent the generation of contamination. We perform atomic-scale AFM imaging and hydration force measurements at a mica–water interface using the fabricated tip and demonstrate its applicability to such an atomic-scale application. With a repeated use of the proposed process, we can reuse a small cantilever for atomic-scale measurements for several times. Therefore, the proposed method solves the two major problems and enables the practical use of a small cantilever in atomic-scale studies on various solid–liquid interfacial phenomena.

Keywords: atomic force microscopy, electron beam deposited tip, atomic-resolution imaging

(Some figures may appear in colour only in the online journal)

1. Introduction

Atomic force microscopy (AFM) [1] is capable of imaging atomic-scale surface structures and properties of various materials, including metals, semiconductors and insulators. It can be operated in liquid, air and vacuum. Owing to the high resolution and wide applicability, the method has been used for various nanoscience research. In particular, liquid-environment applications are rapidly increasing due to recent advancements in AFM instrumentation, as well as growing interests in biological, environmental and energy science.

Recently, the spatial resolution of dynamic-mode AFM in liquid has been significantly improved [2–4], which has enabled subnanometer-scale studies on minerals [2, 5–7], biological systems [8–10] and other organic molecules [11, 12] in liquid. In addition, several three-dimensional (3D)
force measurement techniques based on AFM have been developed [13, 14]. These techniques are used for visualizing 3D distribution of water molecules (i.e. hydration structures) [14, 15] or flexible surface structures [16] at solid–liquid interfaces.

To further expand the capability of atomic-resolution AFM techniques, it is essential to improve their operation speed and force sensitivity. For example, real-time imaging of atomic-scale dynamic processes requires wide bandwidth of the feedback regulation as well as high force sensitivity. Furthermore, high-speed 3D imaging imposes more stringent requirements on the speed and sensitivity due to the large volume of information to be collected.

Operation speed and force sensitivity of AFM are ultimately determined by cantilever characteristics [17, 18]. Thus, significant efforts have been made for improving the cantilever design. To this end, one of the major strategies is reduction of the cantilever size [10, 19–22]. A smaller cantilever gives a higher resonance frequency ($f_0$), which improves AFM operation speed as well as force sensitivity. Recently, AFM probe manufacturers have established a mass production process for small cantilevers with an acceptable yield rate. In the meanwhile, an AFM design that provides the optimal performance of a small cantilever in AFM measurements has also been established [22].

One of the remaining problems in the practical use of small cantilevers is a fabrication method of a small tip. As we reduce the cantilever size, we should also reduce the tip size. However, the conventional tip fabrication method using Si etching process is not necessarily ideal for fabricating such a small tip. As an alternative method, an electron beam deposition (EBD) process of a carbon tip has been studied [23–29]. In the process, a focused electron beam is irradiated at the cantilever end by scanning electron microscope (SEM). This induces deposition of decomposed organic molecules in the SEM chamber and produces a sharp carbon tip.

Although EBD tips have been successfully used for some of the AFM applications [22, 30, 31], their use in atomic-scale measurements in liquid has been hindered by following problems. So far, atomic-scale AFM measurements in liquid has been generally performed with a Si tip [32]. In the meanwhile, an EBD tip is made of carbon. Thus, it is not clear if chemical or mechanical properties of an EBD tip is suitable for such applications. In addition, a high price of a small cantilever is also a serious problem for its routine use.

In this study, we solve these problems to enable routine use of a small cantilever in atomic-scale applications in liquid. We clarify the problems in using as-purchased small cantilevers for such applications. To solve the problems, we propose a fabrication method of an EBD tip suitable for atomic-scale imaging in liquid. The method also enables to reuse a small cantilever for several times. We demonstrate the performance of the fabricated tips by atomic-scale imaging and force measurements at a mica–water interface.

2. Experimental details

2.1. AFM

We used a custom-built frequency modulation AFM (FM-AFM) with an ultra-low noise cantilever deflection sensor [2, 33] and a high stability photothermal excitation system [22, 34]. The use of a photothermal excitation system is generally desirable for stable operation of dynamic-mode AFM in liquid to avoid excitation of spurious resonances of mechanical parts in an AFM system. Furthermore, stable excitation of a small cantilever imposes stringent requirements on the stability of an excitation laser beam. In this study, we used a specially designed photothermal excitation system to achieve the thermal-noise-limited performance of FM-AFM even with a small cantilever.

The AFM head was controlled with a commercially available AFM controller (ARC2, Asylum Research). A cantilever was oscillated at a constant amplitude using a commercially available oscillator system (OC4, SPECS). We performed FM-AFM imaging in the constant frequency shift ($\Delta f$) mode, where tip-sample distance is regulated such that $\Delta f$ is kept constant. FM-AFM imaging and $\Delta f$ curve measurements were performed on a cleaved muscovite mica (01877-MB, SPI Supplies) in 10 mM phosphate buffered saline (PBS) solution (P4417-50TAB, Sigma-Aldrich) at room temperature.

We used commercially available small cantilevers (USC-F5-k30, Nanoworld). An as-purchased cantilever is coated with Au (30 nm) on both the front and back sides. A typical resonance frequency ($f_0$) and Q factor ($Q$) in liquid, and a nominal spring constant ($k$) of the cantilever are 3.2 MHz, 6 and 30 N m$^{-1}$, respectively. A small cantilever comes with an EBD tip with a nominal apex radius and length of less than 10 nm and 2.5 $\mu$m, respectively. For coating a cantilever and tip with Si (30 nm), we used a dc sputter coater (K575XD, Emitech).

2.2. SEM and micro-manipulator

We fabricated an EBD tip by field emission SEM (FE-SEM) (ERA-8000FE, ELIONIX). We irradiated a focused electron beam with a 30 kV acceleration voltage at the cantilever end. We used different irradiation time (20 s-3 min) for fabricating tips with different length. The SEM chamber was pumped with a diffusion pump. Thus, we had sufficient residual gas molecules to produce an EBD tip. For imaging the structure of cantilevers and tips, we used another FE-SEM (JSM-7100F, JEOL). For the imaging, we used a 15 kV acceleration voltage.

We attached a silica bead with a diameter of $\sim$2 $\mu$m (43-00-203 Sicastar, Micromod) with glue (353ND, Epoxy Technologies) using a micro-manipulator (Axis Pro 2: Micro Support). This manipulator was also used for removing an EBD tip.
3. Results and discussions

3.1. Problems in using an EBD tip

3.1.1. SEM images

Figures 1(a) and (c) show SEM images of a conventional cantilever (PPP-NCHAuD, Nanoworld) that has often been used for atomic-scale FM–AFM experiments in liquid. In the meanwhile, figures 1(b) and (d) show SEM images of a small cantilever used in this study (USC-F5-k30, Nanoworld). Nominal dimensions of these cantilevers (length: \( \ell \), width: \( w \), thickness: \( t \), tip height: \( h \)) are shown in table 1. These SEM images and table 1 illustrate the considerable size difference between the two cantilevers.

An NCH cantilever comes with a Si tip fabricated by wet etching. The back side of the cantilever is coated with Au (35 nm) while the tip side is left uncoated. A USC cantilever comes with a sharp carbon tip fabricated by an EBD process. Although both sides of a cantilever are coated with Au (30 nm), the tip surface is left uncoated. According to the manufacturer, the EBD tip is made of high density carbon.

3.1.2. Contaminations

In our previous study [22], we found it difficult to perform atomic-scale FM–AFM imaging with an as-purchased USC cantilever. We also found that this problem can be solved by the Si coating. However, at that time, we were not able to clarify the origins for the difficulty.

Recently, we found that imaging of a mica surface with an as-purchased USC cantilever in PBS solution for a long time results in a gradual increase of surface contaminations. Figure 2(a) shows an FM–AFM image of a mica surface obtained at 40 min after we immersed the cantilever in solution. The image shows that the mica surface is covered with granular contaminations. The observed grains are much larger than an atomic-scale size. We found no clear regularity of their arrangement. These results suggest that an as-purchased USC cantilever produces contaminations in aqueous solution and they are adsorbed on a mica surface. These contaminations may be produced by dissolution of the EBD tip material or desorption of organic molecules deposited on a cantilever surface during the tip fabrication process.

The contamination should be one of the major factors that prevent atomic-scale imaging in liquid. This problem can be solved by the Si coating. We coat a USC cantilever by the sputtering method and hence the whole surface of the cantilever and tip should be covered with Si. This should prevent dissolution of the tip material and desorption of the surface contaminations on a cantilever. Figure 2(b) shows an FM–AFM image of a mica surface obtained using a cantilever with Si coating. The image was taken at 200 min after we

| Table 1. Nominal dimensions of a conventional (NCH) and a small (USC) cantilever. |
|-----------------|--------|-------|-------|--------|
| Cantilever      | \( \ell \) [\( \mu \text{m} \)] | \( w \) [\( \mu \text{m} \)] | \( t \) [\( \mu \text{m} \)] | \( h \) [\( \mu \text{m} \)] |
| NCH             | 125    | 30    | 4.0   | 12.5   |
| USC             | 10     | 5     | 0.7   | 2.5    |
immersed the cantilever in solution. In contrast to the image obtained without Si coating, the image shows clear atomic-scale contrasts. This result demonstrates that the Si coating is effective for reducing the contaminations from the small cantilever.

3.1.3. Mechanical strength. We previously reported that the Si coating enables atomic-scale imaging with a USC cantilever [22]. However, our subsequent experiments revealed that this is not necessarily true for all USC cantilevers. For example, figures 3(a) and (b) show FM–AFM images of a mica surface obtained in PBS solution using different USC cantilevers (USC1 and USC2) with Si coating. The image obtained by USC1 (figure 3(a)) shows atomic-scale contrasts while the one by USC2 (figure 3(b)) shows almost no contrasts. Difference between the two cantilevers is also confirmed in Δf curve measurements. The Δf curve obtained by USC1 (figure 3(c)) shows an oscillatory profile reflecting the hydration structure formed at the mica–water interface. In the meanwhile, the one by USC2 (figure 3(d)) shows a long-range repulsive force instead of an oscillatory one. As a shorter decay length of the force profile is desirable for high-resolution imaging, the results obtained by the force measurements are consistent with those obtained by the imaging. Although the capability of visualizing an oscillatory force profile is not essential for high-resolution imaging, it is desirable for investigating hydration structures at a solid–liquid interface.

The difference between USC1 and USC2 probably originates from different mechanical strength of a tip. The insets of figures 3(a) and (b) show SEM images of the USC1 and USC2 obtained before we coat the cantilevers with Si. The images show that USC2 has a sharper tip than that of USC1. Thus, its mechanical strength may not be sufficient for atomic-scale experiments. The low spatial resolution and the long-range repulsive force can be explained by a large thermal fluctuation of the tip apex position. The latter can also be explained by a deformation of the tip apex caused by a tip-sample interaction force. This argument is further supported by our experiments on the tip length dependence of Δf curves, which is described in section 3.3.

3.2. Fabrication process

3.2.1. Basic concept. We have developed a fabrication process of an EBD tip suitable for atomic-scale imaging in liquid with a USC cantilever. The process also enables to reuse a USC cantilever for several times. Figure 4 shows SEM images of a USC cantilever obtained at each step of the fabrication and reuse process.

An EBD tip that comes with an as-purchased USC cantilever (figure 4(a)) is not necessarily suitable for atomic-scale FM–AFM imaging. Thus, we remove the original tip (figure 4(b)) and fabricate another one. The simplest way of fabricating an EBD tip is to deposit carbon on a cantilever surface. However, this process makes it difficult to satisfy following requirements.

An EBD tip should be long enough to avoid significant Q damping caused by the squeeze film effect [35–37]. In a liquid environment, if an EBD tip is too short, Q significantly decreases when a cantilever is brought close to a sample surface. To avoid this effect, it is ideal to make a tip longer than the cantilever width. Practically, it is difficult to fabricate such a tip with an acceptable throughput. In fact, a USC cantilever comes with a 2.5 μm tip, which is approximately a half of the cantilever width. Using a small electron beam spot size in the EBD process, we can fabricate a long tip in a short time. However, the fabricated tip is typically so sharp that its mechanical strength is insufficient. In the meanwhile, we can use a large beam spot size for fabricating a blunt tip. This, however, requires a long fabrication time. This problem is particularly serious when the process is used for reusing a cantilever or fabricating many tips at a time.
To solve these problems, here we propose following fabrication process. We first attach a silica bead (2 μm) on the cantilever surface and fabricate an EBD tip (500–700 nm) on the bead (figures 4(c) and (d)). The EBD tip is long enough to avoid a significant Q damping as the attached bead provides 2 μm offset in the tip height. The silica bead is fixed with a large contact area, which gives high mechanical strength to the tip.

Compared with the fabrication of an EBD tip directly on a cantilever surface, the proposed method requires an additional process for attaching a silica bead. However, this process is necessary only for the first-time use. In the reuse process, we can remove only an EBD tip and fabricate another tip on the same bead (figures 4(e) and (f)).

Details of the proposed fabrication and reuse processes are individually described in sections 3.2.2–3.2.4.

3.2.2. Removal of an EBD tip. We remove an EBD tip from an as-purchased USC cantilever by the micro-manipulator. Figure 5 shows optical microscope images of a cantilever and an EBD tip obtained during the process. Owing to the high-magnification optics, we can identify the tip position from a side view of a USC cantilever (figure 5(a)). We place a commercially available tungsten probe (TP-0005, Micro Support) above the tip (figure 5(b)). We move the probe downwards to hit the tip and to remove it (figures 5(c) and (d)). The removed EBD tip typically sticks to the tungsten probe. The stuck tip can be removed by a gentle wipe so that we can reuse the tungsten probe. In this way, we can remove an EBD tip as shown in figure 4(b).

3.2.3. Attachment of a silica bead. We attach a silica bead with a diameter of ~2 μm to the cantilever surface with glue using the micro-manipulator. Figure 6 shows optical microscope images of a cantilever obtained during this process. In the process, we use two custom-made glass probes. We drop a small amount of glue on a glass slide and dip a glass probe into the glue so that its apex is covered with it. We bring the other glass probe close to the first one to hold small amount of glue in the gap between the two probes (figure 6(a)). We drop the glue onto the surface of a USC cantilever without an EBD tip (figure 6(b)). We pick up a silica bead by one of the two probes (figure 6(c)) and drop it on the glue deposited on the cantilever surface (figure 6(d)). We transfer the cantilever and heat it at 130 °C for 15 min to set the glue. After the heating, we make sure that the attached bead is not displaced by a gentle push with a clean glass probe. In this way, we can reproducibly fix a silica bead to the cantilever surface as shown in figure 4(c).
3.2.4. Fabrication of an EBD tip. We fabricate an EBD tip on the silica bead attached on a cantilever surface. The growth process of an EBD tip strongly depends on surface properties of the deposition area. Thus, we should control surface condition of a silica bead before the tip fabrication. To this end, we coat a silica bead with Si (30 nm) and immerse the cantilever in acetone for 1 min just before the tip fabrication. After the surface treatment, we attach the cantilever to a custom-made cantilever holder and insert it into the SEM chamber. We irradiate a focused electron beam with a 30.0 kV acceleration voltage to the silica bead. We set the irradiation time at a value between 20 s and 3 min, depending on a desired tip length. In this way, we fabricate an EBD tip on a silica bead as shown in figure 4(d).

The SEM image shows that the EBD tip is tilted from the normal to the cantilever surface. This is because a cantilever is attached to the holder with a 13° tilt from the horizontal plane. This tilt angle agrees with that of a cantilever holder for our AFM. Thus, the fabricated EBD tip is vertically oriented to a sample surface in the AFM setup.

After the tip fabrication, we coat the cantilever and the tip with Si (30 nm) just before using it. This Si coat is effective not only for preventing the contamination but also for improving the stability and reproducibility in atomic-scale AFM measurements in liquid [38].
Figure 5. Optical microscope images of a cantilever and an EBD tip obtained during a tip removal process.

Figure 6. Optical microscope images of a cantilever obtained during an attachment process of a silica bead to the cantilever surface.
After an AFM experiment in liquid, we immediately immerse the used cantilever in pure water without exposing it to the air. Otherwise, salts can precipitate on the cantilever surface. Once this happens, we found it difficult to remove the precipitated salts by a simple surface treatment method.

When we reuse a cantilever, we remove an EBD tip in the same way as described above (figure 4(e)). After the removal of an EBD tip, we again coat a cantilever with Si (30 nm) and fabricate an EBD tip by FE–SEM. Figure 4(f) shows an SEM image of an EBD tip fabricated for the second time. By repeating these processes, we can reuse a cantilever for several times.

Repeated use of a cantilever results in an increase of the deposited Si on the cantilever surface and hence an increase of $f_0$, $Q$ and $k$. In addition, an experiment in a solution containing various solutes can result in serious contamination of the cantilever surface. These factors practically determine the maximum reuse cycles. At present, we can typically reuse a cantilever for ~5 times. This may be increased by reducing the thickness of a Si coat.

### 3.3. Performance of an EBD tip

We have investigated performance of an EBD tip fabricated by the process described above in atomic-scale AFM measurements in liquid. We performed FM–AFM imaging and $\Delta f$ curve measurements on a mica surface in PBS solution with different tip length (figures 7(a)–(h)).

For a tip shorter than 300 nm, we can obtain atomic-scale FM–AFM images with a relatively high $\Delta f$ set point (>20 kHz) as shown in figure 7(a). However, we found that atomic-scale imaging with a low $\Delta f$ set point is often difficult. This is because of the gradual increase of a $\Delta f$ signal during the tip approach (figure 7(b)). This long-range force probably originates from the interaction between the bead and sample surface. With decreasing the tip length, the bead-sample distance after the tip approach becomes shorter. Thus, the contribution from the interaction between them should become evident. The large $\Delta f$ set point results in a large loading force during the imaging. This may prevent gentle imaging of soft materials. Therefore, applicability of such a short tip is limited.

With a tip longer than 800 nm, we were not able to obtain a clear atomic-scale image (figure 7(g)). The $\Delta f$ curve does not show an oscillatory profile but only a gradual increase as the tip approaches (figure 7(h)). This long-range force typically has a much longer decay length (~0.33 nm) than that with a short tip (~0.14 nm) (figure 7(b)). A long tip with a high aspect ratio has a relatively low mechanical strength. This results in a large thermal fluctuation, and tip deformation caused by the interaction with a sample surface. Such fluctuation and deformation lead to an increase of a long-range repulsive force and hinders atomic-scale AFM experiments. This argument is consistent with the discussion on the as-purchased USC cantilever described in section 3.1.3.

With a tip between 500–700 nm, we can reproducibly obtain atomic-scale FM–AFM images. The $\Delta f$ curve does not show a gradual increase but an oscillatory profile reflecting the hydration structure formed at the interface. The result shows that the tip is long enough to suppress the influence from the interaction between the bead and sample surface. The results shown in figures 7(c) and (d) were obtained with the second tip fabricated on the cantilever. Similar results were also obtained with the second tip fabricated on the same cantilever as shown in figures 7(e) and (f). These results demonstrate that the reused cantilever is also applicable to...
atomic-scale AFM measurements as long as the tip length is within this range.

For quantitative discussions, we approximately calculated the bending stiffness \(k_t\) and thermal vibration amplitude \(\delta x\) of an EBD tip. Here we assume that an EBD tip is a circular truncated cone with diameters at the fixed and free ends of \(a_1\) and \(a_2\), respectively. With this model, \(k_t\) and \(\delta x\) are given by

\[
k_t = \frac{3\pi E}{64} \int_0^\ell \int_0^s \frac{\ell - s}{\{a_1 - (a_1 - a_2)(s/\ell)^4\}^4} ds dx, \tag{1}\]

\[
\delta x = \sqrt{k_B T/k_t}, \tag{2}\]

where \(\ell\), \(E\), \(k_B\) and \(T\) denote length, modulus, Boltzmann’s constant and temperature, respectively. According to previous studies [39] and information obtained from the probe manufacturer, \(E\) of a carbon nanopillar is typically in the range of 100–800 GPa. With \(a_1 = 120\text{ nm}\) and \(a_2 = 20\text{ nm}\), we calculated \(\delta x\) for each tip length as described in the caption for figure 7. For \(\ell = 700\text{ nm}\), we obtain \(k_t = 1.5–11.9\text{ N m}^{-1}\) and \(\delta x = 18–53\text{ pm}\). This result suggests that \(\delta x\) should be less than \(\sim 50\text{ pm}\) for visualizing the periodic structure of a mica surface (\(\sim 500\text{ pm}\)). Although this is a rough estimate, the obtained values sound reasonable.

The tip length required for suppressing the bead-sample interaction may depend on the bead size. However, we were not able to investigate this dependence due to the limited size range of a bead that we can use for this experiment. The minimum size that we can manipulate with our micro-manipulator is \(\sim 2\mu \text{m}\) while the cantilever width for the tip side is \(\sim 3\mu \text{m}\). Within such a limited range, we speculate that the influence of the size variation should not be significant.

4. Conclusions

In this study, we have clarified major problems in using a small cantilever with an EBD tip for atomic-scale AFM measurements in liquid. To solve these problems and to enable practical use of a small cantilever, we have developed a fabrication process of an EBD tip suitable for such applications.

Immersion of an as-purchased small cantilever in aqueous solution leads to generation of contaminations from the cantilever and/or tip surface. In addition, owing to a high thermal fluctuation and deformation, these problems often prevent applications of a small cantilever to atomic-scale measurements in liquid.

To resolve these problems, we have proposed a fabrication process of an EBD tip, where we attach a 2 \(\mu\)m silica bead at the end of a cantilever and fabricate a 500–700 nm EBD tip on the bead. The bead height ensures sufficient cantilever-sample distance and enables to suppress long-range interaction between them even with a short EBD tip having high mechanical strength. After the EBD tip fabrication, we coat the whole cantilever and tip surface with Si (30 nm) to prevent generation of contaminations as well as to make the tip surface condition suitable for atomic-scale imaging in liquid.

To investigate the applicability of the fabricated tip to atomic-scale AFM measurements in liquid, we performed FM–AFM imaging and \(\Delta f\) curve measurements on a mica surface in PBS solution using tips with different length. With a tip shorter than 300 nm, the long-range force acting between the bead and sample surface becomes evident and hinders high-resolution imaging with a low \(\Delta f\) set point. With a tip longer than 800 nm, thermal fluctuation and deformation of the tip apex prevent high resolution measurements as in the case of an as-purchased small cantilever. We can solve these problems using a tip with a moderate length: 500–700 nm. As long as the tip length is in this range, a reused cantilever is also applicable to atomic-scale measurements in liquid.

So far, significant advantages of using a small cantilever in atomic-scale AFM measurements in liquid have been well demonstrated [10, 22]. Nevertheless, its routine use in practical applications has been hindered by the tip issues and the high cost. The method proposed in this study solves these major problems and enables practical use of a small cantilever. In addition, the significant improvements in the operation speed and force sensitivity achieved by a small cantilever should open up possibilities for further development of advanced AFM techniques such as high-speed, 3D and multi-frequency AFMs. These technical advancements should provide powerful means for atomic-scale studies on various solid–liquid interfacial phenomena.

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