Low Energy Oxygen Ion Beam Machining of Ultra Smooth and Sharp AFM Nano-Tips from Single Crystal Diamond Rods

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Diamond made AFM probe exhibits a long life time as diamond has ultrahigh hardness, ultra high tensile strength and high wear resistance. For the ultra fine machining of diamond tip, focused ion beam (FIB) machining is being widely used but the process is very slow due to one by one processing and ripples are formed on the processed surface. Moreover, the irradiation damage and machining cost are significantly high. Compared to FIB machining, low energy broad ion beam machining (BIBM) is better suitable for the mass fabrication of AFM tips. In this paper, we presented a two stage BIBM process using 1-3 keV O\(^+\)/O\(_2\)\(^+\) ion beam for the mass fabrication of ultra smooth and sharp AFM nano-tips from single crystal diamond rods. In order to suppress the ripple formation on the processed diamond tip, the sample was rotated during machining process. In our proposed method, around 100 ultra smooth and sharp AFM nano tips with the apex angle of 50° and an average tip diameter of 22 nm can be simultaneously fabricated in just 1.5 hour. Moreover, the irradiation damage of the processed tip is also very low due to low energy ion beam machining and chemical etching of the amorphous carbon by the oxygen ions. A simulation model is developed to predict the profile change of diamond tip by low energy oxygen ion beam machining. At last, the mechanisms of fine sharpening and ultra smoothening of the diamond tips are discussed with the help of Witcomb formula and Bradley-Harper (BH) model. [DOI: 10.1380/ejssnt.2012.447]

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I. INTRODUCTION

Monolithic silicon AFM tip, an integral part of a cantilever, can be mass-produced using a semiconductor manufacturing process. However, the life time of the Si tips is very short due to lower hardness and poor wear resistance. Therefore, Si made AFM probes cannot be applied for large area measurement. Compared to Si, diamond exhibits many superior properties such as ultra-high hardness, ultrahigh tensile strength and high wear resistance [1–8]. Therefore, a high-aspect ratio AFM probe made of diamond is very promising for deep trench measurement, repeated scanning and probe based manipulation at the atomic level. Conventionally, diamond nano-tip for AFM probes are fabricated using a combination of CVD-diamond film, standard lithography and dry etching process [9–12]. Among the dry etching processes, PE (plasma etching) [13], RIE (reactive ion etching) [14, 15], IBM (ion beam machining) [16–18], RIBM (reactive ion beam machining) [18], IBAE (ion beam assisted etching) [19], RIBM (reactive ion beam machining) and FIB (focused ion beam) machining are being widely used [20, 21] for ultra-fine machining of diamond tools. RIE, which is commonly used for surface micro-machining of CVD diamond MEMS, cannot be applied for the forming and sharpening of diamond probes used in AFM because in this method the incident angle of the ions cannot be changed as required, hence it becomes difficult to control the shape of the diamond tip. FIB machining method can be employed for the sharpening of diamond probe [22], and for the forming of diamond micro-tools [23, 24]. However, this process also has some drawbacks such as formation of ripples on the probe’s surface and high radiation damage due to high operating energy (usually 40-50 keV). Moreover, the process is slow (due to one by one processing) and running cost is high. The limitations of FIB machining can be eliminated to a great extent by applying low energy broad ion beam machining (BIBM). BIBM was applied by Nagase et al. for the simultaneous fabrication of AFM nano-tips from single crystal diamond rods by 3-10 keV He\(^+\), Ar\(^+\) and Xe\(^+\) ion beam [25]. However, in this method, the machining time was quite long (around 6 hrs). Moreover, ripples were found on the fabricated tips in case of Ar\(^+\) and Xe\(^+\) beam which is unacceptable for high accuracy and ultra-precision level measurement by AFM. In order to improve the machining speed and ensure least irradiation damage, we introduced here a two step RIBM method using low energy (1-3 keV) O\(^+\)/O\(_2\)\(^+\) ion beam. In our proposed method, many (about 100) ultra smooth and sharp AFM nano-tips (apex angle of \(\approx\)50°, tip diameter 20-25 nm) were simultaneously fabricated at a much shorter processing time < 2 hrs. The diameter and apex angle of the processed and unprocessed tips were measured from the scanning electron microscope (SEM) photograph (we defined as experimental result). We also developed a simulation model to predict the profile change of diamond tip by low energy oxygen ion beam. The simulation result is compared with the experimentally obtained profile.

The formation of ripple on the machined surface was avoided by machining under rotating condition. The effect of rotation on the surface roughness was examined on
a flat diamond substrate by an atomic force microscope (AFM). Finally, the mechanism of ultra smoothening of the diamond tips is discussed with the help of Bradley-Harper (BH) model [26] and then compared with the theoretically derived apex angle using Witcomb formula.

II. EXPERIMENTAL APPARATUS

The experiments were conducted in an electron cyclotron resonance (ECR) type ion beam apparatus which can generate broad ion beam in the energy range of 0.3-3 keV. The pressure inside the plasma generation chamber was set at $1 \times 10^{-4}$ Pa. Figure 1 shows the ECR type ion beam apparatus. Gas was passed into the plasma generation chamber, ion was formed inside the chamber and then the samples were sputtered under rotating condition. The measurement of etching depth of natural single crystal diamond chip (100) was done by Alpha-Step 500 surface profiler. The un-processed and processed diamond rods were observed by a SEM. The Simulation of the profile change was developed by Matlab software. In order to evaluate the effect of rotation on diamond surface, a diamond chip of uniform surface roughness $1.0 \pm 0.12$ nm rms was taken as a sample, segmented by tapes and machined different segments by $O^+ / O_2^+$ beam with and without rotation at 3 keV ion energy for 1/2 hr. The surface roughness was measured by AFM before and after the ion beam machining.

III. RESULTS AND DISCUSSIONS

A. Experimental observation

In order to measure the etching rate, the natural single crystal diamond chips (100) were sputtered by 1 keV and 3 keV oxygen ion beam at different angles of ion incidence. The ion current densities were 1.1 mA/cm$^2$ (for 1 keV) and 1.6 mA/cm$^2$ (for 3 keV) during the etching process. The etching depth of natural single crystal diamond chips was measured by Alpha-Step 500 surface profiler. Figure 2 shows the plot of etching rate, $V$ ($\mu$m/h) at different angles of ion incidence ($\theta$) for both 1 keV and 3 keV $O^+ / O_2^+$ beam. During the oxygen ion beam etching process, both chemical sputtering and physical sputtering takes place and chemical sputtering is more active towards lower ion energy and lower ion incidence angle [27]. As shown in Fig. 2, the etching rate is found higher at 1 keV ion energy compared to that at 3 keV due to dominance of chemical sputtering over physical sputtering in the former case.

In the next stage, we carried out the fabrication process of AFM nano-tips by $O^+ / O_2^+$ beam at both 1 keV and 3 keV ion energy. At first, many natural single crystal diamond rods of 100 $\mu$m length and 2 $\mu$m diameter were fabricated in a work piece by a metallographic etching technique using Ni film [28, 29]. The fabricated diamond rods are shown in Fig. 3. Then, the work piece with around 100 diamond rods was placed on the stage of ion beam processing chamber. The sharpening of the diamond rods were performed at normal angle of ion incidence by different energies of $O^+ / O_2^+$ beam. During the machining process, the stage was rotated.

Figures 4(a) and (b) show the SEM images of the processed diamond rods by 1 keV and 3 keV $O^+ / O_2^+$ ion beam respectively. As shown in Fig. 4(a), the apex angle and tip diameter of the processed rods at 1 keV were 66$^\circ$ and 50 nm respectively. The tip became sharper by 3 keV oxygen ion beam. As shown in Fig. 4(b), the obtained apex angle and tip diameter were 50$^\circ$ and 22 nm respectively at 3 keV. The desired specifications of the nano-AFM tips (apex angle $\leq 50^\circ$, tip diameter 20-25 nm) were attained by 3 keV $O^+ / O_2^+$ beam machining process though it took a longer machining time (2.5 hrs) than that of 1 keV ion energy (1.5 hrs) due to slower etching rate. In order to fasten the machining speed further, we proposed a two stage machining process of the diamond tips. In our proposed method, the 1 keV and 3 keV $O^+ / O_2^+$ ion beam machining processes were combined i.e at first ma-
Fig. 4: SEM images of processed diamond rods (a) by 1 keV O⁺/O₂⁺ beam, and (b) by 3 keV O⁺/O₂⁺ beam.

Fig. 5: SEM images of the diamond rods at different stages of 1-3 keV O⁺/O₂⁺ beam machining (a) un-processed rod (b) processed rod by 1 keV O⁺/O₂⁺ beam after 0.5 hour, (c) processed rod by 1 keV O⁺/O₂⁺ beam after 1 hour, (d) finally processed rod by 3 keV O⁺/O₂⁺ beam after 1.5 hour. $t$ indicates the time from starting of the combined machining process.

Machining was done by 1 keV O⁺/O₂⁺ beam for 1 hour and then the ion energy was increased to 3 keV and processed further for 0.5 hour. The result of the combined machining process is represented in Fig. 5. Figure 5(a) shows the SEM image of an unprocessed single crystal diamond rod. From this image, we can see that the unprocessed surface was very rough with a lot of protrusions and irregularities present both in regions near the head and sidewall. Figure 5(b) shows the image of the diamond rod after partial machining done by 1 keV O⁺/O₂⁺ beam for 0.50 hr. It is found that the protrusions on the surface of the diamond rods were diminished after partial machining but the head was blunt. Therefore, we extended the sharpening time. The rods turned smoother and sharper after 1 hour of machining under the same condition which is shown in Fig. 5(c). In the second stage, the machining was done at 3 keV oxygen ion energy for 0.5 hour and the diamond rods became completely smooth and sharp at the end without forming any ripples. Figure 5(d) shows the SEM image of a finally processed ultra smooth and sharp diamond rod of apex angle 50° with a tip diameter of 22 nm which is suitable to be used as an AFM nano-tip. The required total machining time of our proposed method was only 1.5 hrs which is 4 times shorter than that of 3-10 keV He⁺, Ar⁺ and Xe⁺ ion beam machining [25].

Another significant factor need to account during IBM is the irradiation damage. When a diamond surface is bombarded by ion beam, a damage layer is induced by the physical sputtering and the diamond crystal structure changes into amorphous carbon or graphite. Physical sputtering becomes more pronounced as the ion energy increases causing higher irradiation damage. Kawabata et al. identified the irradiation damage of different ion beam etching process by the shift of binding energy from 285.2 eV (diamond) to 284.2 eV (graphite) and broadening of the peak in the XPS profile [30]. They reported that RIBAE and RIBM have lower tendency to form graphite than IBE and IBAE. It is because in RIBAE and RIBM, chemical sputtering takes a significant role and it becomes more pronounced towards lower ion energy. Therefore, in case of oxygen ion beam processing, the physical sputtering induced damage structure on the diamond surface is simultaneously etched out by the chemical sputtering creating volatile substances like CO₂ or CO [31]. Hence, the diamond composed base layer is recovered mostly by the RIBM.

B. Simulation of the profile changes by 3 keV O⁺/O₂⁺ beam machining ($t = 0.5$ hr)

The simulation of the profile change from partially processed diamond rod to finally processed sharp diamond tip by 3 keV O⁺/O₂⁺ beam machining is developed in the following manner.

At first, it is assumed from Fig. 5 that the profile takes a curved shaped after 1 hr machining by 1 keV O⁺/O₂⁺ beam. Then, it is considered that the profile is divided
into ‘i’ number of segments. Due to curved shape of the profile, the ion incidence angle on each segment will be different and hence the etching rate. Therefore, after time \(T\), the etching depth reached by each segment of the diamond rod can be presented by Eq. (1):

\[
R_i = V_i \cdot T.
\]

Here, \(R_i\) and \(V_i\) represent the vector of etching depth and etching rate respectively corresponding to ith segment. Now we consider an initial point \(P(x_0, y_0)\) on the curved surface at vertical angle \(\theta\) will move to a new point \(P(x, y)\) due to ion beam machining for \(t\) hours which can be expressed by the following equations according to Ducemond et al. [32]:

\[
x = x_0 - \left( \frac{dV(\phi)}{d\phi} \cos \phi + V(\phi) \sin \phi \right) t,
\]

\[
y = y_0 - \left( \frac{dV(\phi)}{d\phi} \sin \phi + V(\phi) \cos \phi \right) t.
\]

The simulation of the profile change at the final stage of machining by 3 keV O\(^+\)/O\(_2\)\(^+\) beam for 1/2 hr is shown in Fig. 6. The profile at \(t_0\) represents the curved shape of the partially processed diamond rod by 1 hr of 1 keV O\(^+\)/O\(_2\)\(^+\) beam machining. The profiles at \(t_1\) and \(t_2\) represent the shape of the processed rod by 3 keV O\(^+\)/O\(_2\)\(^+\) beam machining for 15 min and 30 min respectively. As shown by the profile change from \(t_0\) to \(t_2\), it is realized that the diamond rods become significantly sharp and reach the apex angle of 50° after 1/2 hr machining by 3 keV O\(^+\)/O\(_2\)\(^+\) beam machining at the final stage. Comparing Figs. 5 and 6, it is apparent that the profile change derived from simulation is in good agreement with the experimental observation.

C. Theoretical analysis

1. Theoretical derivation of apex angle from Witcomb formula

Due to irregular shape of the unprocessed diamond rod, the ion incidence angle on each point on the rod will be different. The etching rate depends on ion incidence angle, ion energy and ion species. Therefore, the apex angle of the processed rod also becomes different for different ion species and ion energy. Theoretically, the value of the apex angle, \(\alpha_f\) can be predicted by the Witcomb formula [33] by the following equations:

\[
\theta_{\text{max}} = 90° - \frac{K}{2d(Z_1^{2/3} + Z_2^{2/3})^{1/2}} \times \left( \frac{Z_1 Z_2 (Z_1^{2/3} + Z_2^{2/3})^{2/3}}{n^2 E} \right)^{1/4}
\]

\[
\alpha_f = 180° - 2\theta_{\text{max}},
\]

where \(\theta_{\text{max}}\) is the ion incidence angle where etching rate is the maximum, \(Z_1\) and \(Z_2\) are the atomic numbers of bombarding ion and the target atom, respectively, \(E\) (eV) is the ion energy, and \(n\) (atoms/angstrom) is the atomic density of the target and \(K\) is the fitting parameter.

If the fitting parameter \(K\) is chosen to be 210, the theoretically derived \(\alpha_f\) becomes 50° for \(E = 3\) keV which match with the experimental result. From the formula, it is found that the apex angle becomes smaller at higher ion energy though the change in apex angle is minor for ion energy > 3 keV. Hence, if sharpening of the tip is needed beyond 50°, machining can be done at higher ion energy up to 5 keV. However, in this case reactive ion beam assisted etching (RIBAE) using oxygen as the assist gas and ion species is better suggested in order to ensure least amount of irradiation damage and fast machining process [30].

From the Witcomb formula, it is also realized that the apex angle is related to the atomic number of the bombarding ion and the lower is the atomic number, the lower is the apex angle. Hence, He\(^+\) beam machining gives sharper tip than our proposed method though it takes much longer machining time [25]. Therefore, if acute sharpening of the tip is needed (<40°), the processed rod obtained in our method can be treated further under He\(^+\) beam at 3 keV ion energy. However, too sharp AFM tip is not always recommended since it may create scratches on the surface.

2. Comparative analysis on the evolution of surface morphology of the processed tip

Smoothness of the processed surface is essential for high accuracy measurement done by AFM tips. In our proposed method, the surface was completely smooth which is in contrast with the experimental result of Nagase et al. The discrepancy may be explained in term of BH model. The evolution of ripple patterns and roughening at off-normal ion incidence angle was explained by Bradley and Harper et al. [26] by the following equation:

\[
\frac{\partial h}{\partial t} = -V_0(\theta) + \frac{\partial V_0(\theta)}{\partial \theta} \frac{\partial h}{\partial \theta} + \mu_x \frac{\partial^2 h}{\partial x^2} + \mu_y \frac{\partial^2 h}{\partial y^2} - b \nabla^4.
\]

Here, \(V_0(\theta)\) is the erosion rate of the unperturbed flat surface at an angle \(\theta\) between the surface normal and incident ion beam direction, the terms \(\mu_x\) and \(\mu_y\) are effective surface tension coefficient caused by the curvature dependence of the surface erosion process which leads to surface roughening. The coefficient \(B\) is related to the smoothing mechanisms such as surface diffusion (SD) mediated smoothing, surface erosion smoothing (SES) which mimics surface diffusion and the ion enhanced viscous flow relaxation (IVF) smoothing [34].

In the region near the head of the tip where ion incidence angle is comparatively lower, the dominant smoothing mechanism is uncertain. In Bradley and Harper’s original exposition of the model, \(B\) is formally a thermally activated surface diffusion rate. However, this is unlikely to be the dominant smoothing mechanism in this case of a very high cohesive energy for diamond and low temperature. Umbach, Headrick, and Chang [34] have shown that viscous relaxation in a thin layer of amorphous material near the surface with depth on the order of the ion range, has the same spatial frequency dependence as a surface diffusion process. Assuming the relaxation process is thin film viscous relaxation, as per Umbach [35]...
the relaxation rate is related to surface viscosity by

$$ B = a^3 \gamma / \eta_s, \quad (7) $$

where $a$ is the ion range, $\gamma$ is the surface tension, $\eta_s$ is the surface viscosity. The ion range, $a$ of Ar$^+$, He$^+$ and O$^+$ at 3 keV ion energy for different ion incidence angles is enlisted in Table I which is calculated by TRIM.

It is obvious from Table I that the order of ion range is He$^+ > O^+ > Ar^+$ for the entire range of incidence angle. Hence, the smoothing mechanism related to surface confined viscous flow is found stronger in He$^+$ and O$^+$ compared to that in case of Ar$^+$. Therefore, it can be assumed that it is due to surface confined viscous flow related smoothing mechanism that makes the rough surface smooth in region near the head of the diamond rod when processed by He$^+$ and O$^+$ beam.

Another important technique of suppressing ripple formation where ion incidence angle is high is rotation of the sample during ion beam machining. According to Bradley and Cirlin et al. [36], when the sample is rotated, the smoothing effects of viscous flow and surface self-diffusion may prevail over the roughening effect of the curvature-dependent sputter yield and generate a smooth surface. Though literally, sample rotation is no surface relaxation process in the strictest sense. However, due to the rotation of the sample around its surface normal, the strength of the curvature dependent sputtering can be reduced and the preferred azimuthal direction given by the incident ion beam is lost, thus allowing only isotropic surface structures to appear. Nevertheless, in many cases sample rotation does not always suppress surface roughening but often the surfaces roughen with a slower rate compared to the case for no sample rotation [36–38].

In order to realize the effect of sample rotation, we conducted experiment on flat diamond surface by 3 keV O$^+$/O$_2^+$ beam for 1/2 hour varying the ion incidence angle. The experimental result is represented in Fig. 7. The AFM image of the unprocessed surface of roughness, $R=1.0 \pm 0.12$ nm rms is shown in Fig. 7(a). Figure 7(b1) shows the AFM image of the processed surface when machining was done at 60$^\circ$ ion incidence angle without rotating condition. It is found that ripples were formed and the surface roughness, $R$ increases to 1.33$\pm$0.04 nm rms. On the other hand, when machining was done under rotating condition at the same ion incidence angle ($\theta=60^\circ$), ripples did not form and surface roughness, $R$ decreased to 0.52$\pm$0.03 nm rms which is shown in Fig. 7(b2). Therefore, it can be said that sample rotation eliminated ripple formation in our experiment. The sample was also machined at $\theta = 75^\circ$ with and without rotating condition in order to realize the smoothing mechanism on the side walls of the diamond tips where ion incidence is close to grazing incidence. The results after 1/2 hr machining are shown in Figs. 7(c1) and (c2). It is found that the surface became quite smooth in both cases and the smoothness was greater in case of machining under rotating condition. The surface roughness, $R$ of the machined sample with and without rotation were $0.21\pm0.08$ nm rms and $0.36\pm0.03$ nm rms respectively. The reason of surface smoothing at the grazing incidence by O$^+$/O$_2^+$ beam may be attributed to enhanced erosion of the surface protrusions. Usually at grazing incidence, most of the ions incident will be reflected away after collisions with surface protrusions. This situation changes if the ion impinges near surface steps or similar surface irregularities, where the probability of sputtering off atoms from the surface is significantly increased [38]. Moreover, grazing incidence on the surface is equivalent to near normal incidence on the surface protrusions. As reported by Miyamoto et al. [27], chemical sputtering is more strong towards near normal incidence and physical sputtering becomes dominant at high incidence angles. Therefore, in case of O$^+$/O$_2^+$ beam processing, the protrusions from the side walls are etched out by the chemical sputtering effect of the O$^+$/O$_2^+$ leading the surface become smooth. The smoothing effect.

### Table I: Ion range of Ar$^+$, He$^+$ and O$^+$ at 3 keV ion energy for different ion incidence angles.

| Angle of ion incidence | $Ar^+$ | $O^+$ | $He^+$ |
|------------------------|--------|-------|--------|
| $0^\circ$              | 52     | 75    | 267    |
| $30^\circ$             | 46     | 66    | 247    |
| $45^\circ$             | 37     | 54    | 212    |
| $60^\circ$             | 27     | 42    | 170    |
| $80^\circ$             | 15     | 29    | 130    |

![Fig 7](http://www.sssj.org/ ejsnsnt)
was enhanced under machining with rotation. While in case of He\(^+\)/Ar\(^+\)/Xe\(^+\) beam, the reflection of the ions from the surface at the grazing incidence angle is more dominant than enhanced erosion of the protrusions, therefore, the side walls remained rough in their case. From the experiment on flat diamond surface, it is realized that sample rotation contributed to smoothening of the surface of the diamond rods.

### IV. CONCLUSIONS

In this paper, we presented low energy (1-3 keV) O\(^+/O_2^+\) beam machining (RIBM) for the fabrication of multiple numbers of AFM nano-tips from single crystal diamond rods. Our proposed method consists of two stages. In the first stage machining is done by 1 keV O\(^+\) beam for 1 hour. Then in the second stage, further machining is done on the processed rods by 3 keV O\(^+/O_2^+\) for 0.5 hour. From our experiment and observation, we can draw the following conclusions.

1. The obtained apex angle and tip diameter of the fabricated AFM tip by our proposed method are 50° and 22 nm respectively.

2. The total machining time of our proposed method is 1.5 hour which is 4 times faster than the broad ion beam machining (BIBM) using 3-10 keV He\(^+\), Ar\(^+\) or Xe\(^+\).

3. The O\(^+/O_2^+\) beam reactive sputtering exhibits least amount of irradiation damage on the diamond crystal structure because the amorphous carbon is chemically etched out by oxygen. The irradiation damage is also low due to machining in the low ion energy range (1-3 keV).

4. In case of sharpening needed beyond 50°, 3 keV He\(^+\) beam machining may be applied at the final stage. Another alternative is to machine the unprocessed diamond rods by RIBAE process using oxygen as the ion and assist gas species at higher ion energy (up to 5 keV).

5. Unlike He\(^+\), Ar\(^+\) and Xe\(^+\) beam machining process, the fabricated rods in our method were completely smooth and ripple free.

6. We consider that the surface confined viscous flow and the beneficial effect of sample rotation are the two dominant reasons of smoothening in areas near the head of the processed tip. The reason of smoothening on the side walls of the processed rods by O\(^+/O_2^+\) beam machining process may be attributed to enhanced erosion of the surface protrusions by the chemical sputtering of oxygen ion.

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