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# Nanomachining of Fused Quartz Using Atomic Force Microscope

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<http://dx.doi.org/10.5772/64408>

## Abstract

Nanomachining experiments on fused quartz surface have been performed using an atomic force microscope combined with a two-axis capacitive force/displacement transducer. The minimum normal force  $f_{nP}$  needed to form reproducibly a groove was about  $4.7 \mu\text{N}$ . The minimum critical normal force  $f_{nR}$ , tangential force  $f_{tR}$ , and groove depth  $d_{gR}$  when the material removal process began were found to be  $33.7 \mu\text{N}$ ,  $18.7 \mu\text{N}$ , and  $4.3 \text{ nm}$ , respectively. Characteristic changes in the swelling ratio  $R_s$  and the ratio of force components at the critical normal force  $f_{nR}$  can be used to identify the critical condition for changing from plastic deformation to material removal process region.

**Keywords:** nanomachining, atomic force microscope, material removal process, fused quartz, three-sided pyramidal diamond tip

## 1. Introduction

Recently, surface modifications with a scanning probe microscope have attracted particular interest as a nanomachining technique for processing surfaces on a nanometer scale [1–4]. Especially, nanomachining methods using an atomic force microscope (AFM) will play an increasingly important role in an ultrafine fabrication technology to produce various Nano/microdevices regardless of conductivity [5–18]. However, most of the previous studies on nanomachining have been carried out using a cantilever-type AFM [19–25]. In this AFM, the probe tip as a machining tool is asymmetrically supported by the cantilever. Therefore, because of the asymmetric deflection of the cantilever, the tool tip cannot contact the work surface symmetrically and accurately. To analyze the precise mechanism of nanometer-scale machining, it is not suitable to use such a cantilever-type AFM.

The objective of this chapter is to clarify the fundamental mechanisms of material removal in nanomachining using an AFM diamond tip. To investigate the critical conditions needed to initiate the material removal process, such as the minimum normal force, tangential force, and groove depth when the removal process begins, a series of nanomachining experiments on polished quartz surfaces was conducted using an AFM combined with a two-axis capacitive force/displacement transducer [26–30].

2. Experimental procedure for nanomachining

**Figure 1** shows the experimental method for nanomachining used in this study and its cross-sectional machining model is illustrated in **Figure 2**. By means of the AFM diamond tip as a machining tool, the straight nanometer-scale grooves were machined under a constant normal machining force  $f_n$  and a constant machining speed  $v_m$ . The tangential machining force  $f_t$  was measured experimentally and was used to elucidate the material removal mechanism in nanomachining.

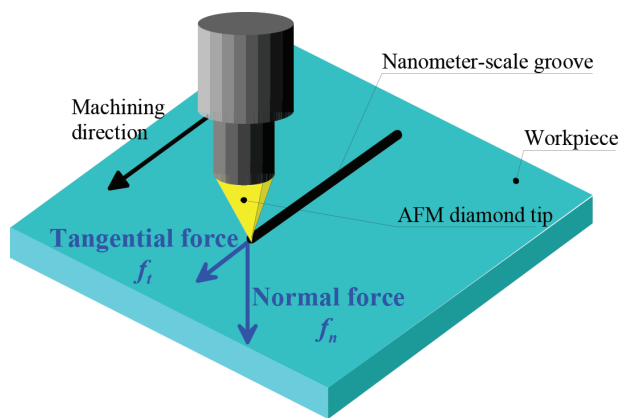


Figure 1. Experimental method for constant-force nanomachining.

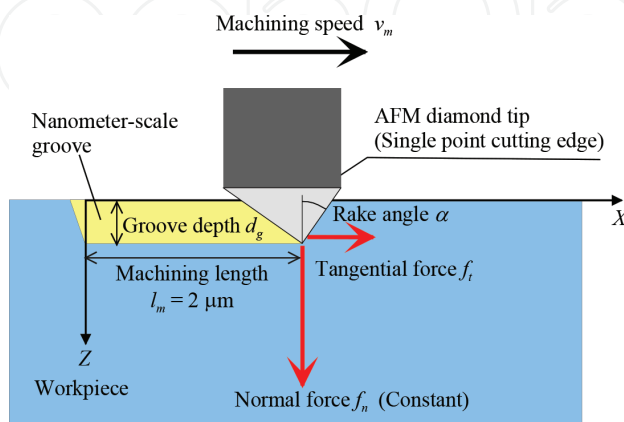
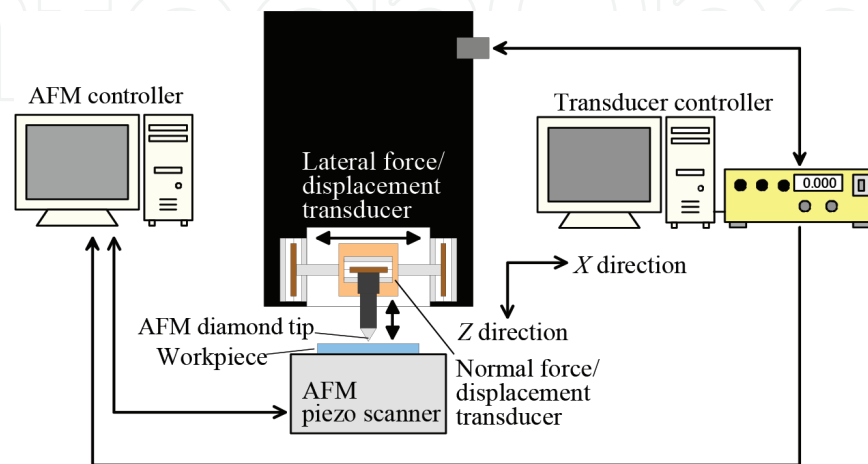


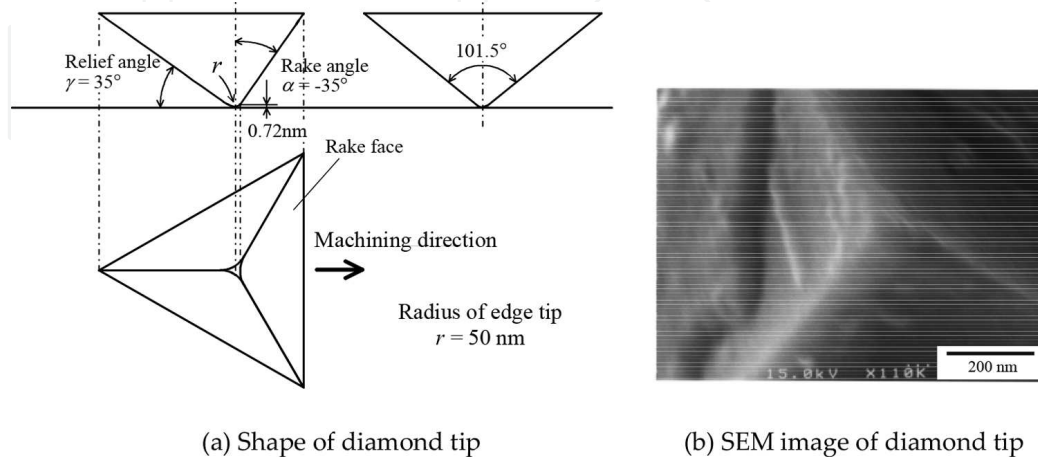
Figure 2. Cross-sectional machining model for constant-force nanomachining with AFM diamond tip.

Nanomachining experiments on fused quartz were performed using an AFM combined with a two-axis capacitive force/displacement transducer, a schematic illustration of which is shown in **Figure 3**. The  $x$ -axis transducer (lateral force transducer) is comprised of two additional sensors that are mounted transversely to the  $z$ -axis force transducer (normal force transducer). Using this two-axis transducer, the force and the displacement in both the  $z$ - and  $x$ -axes were measured.



**Figure 3.** Schematic illustration of constant-force nanomachining equipment with AFM.

A three-sided pyramidal diamond tip (cube corner tip) with a radius of 50 nm set symmetrically in the center of the z-axis force transducer was used for both nanomachining and imaging in these experiments. The shape of the diamond tip, the SEM image of its top edge, and the machining direction used for processing straight grooves are shown in **Figure 4**. Experimental conditions for processing straight grooves are shown in **Table 1**. After the nanomachining experiments, the modified surface was immediately imaged at a normal force of about 0.5  $\mu\text{N}$  using the same diamond tip.



**Figure 4.** Dimensions of AFM tip and its SEM image. (a) Shape of diamond tip. (b) SEM image of diamond tip.

Machining tool	Three-sided pyramidal diamond tip Tip edge radius: 50 nm Rake angle: $-35^{\circ}$ Relief angle: $35^{\circ}$
Machining length $l_m$	2 $\mu\text{m}$
Machining speed $v_m$	100 nm/s
Nominal normal force $F_n$	1, 2, 4, 6, 8, 10, 20, 30, 40, 50, 60, 70, 80, 90, 100 $\mu\text{N}$
Work material	Fused quartz Nanohardness: 8.7 GPa Elastic modulus: 72 GPa Surface roughness: 0.5 nmRa
Atmosphere	In air Temperature: $22^{\circ}\text{C}$ Humidity: 50%

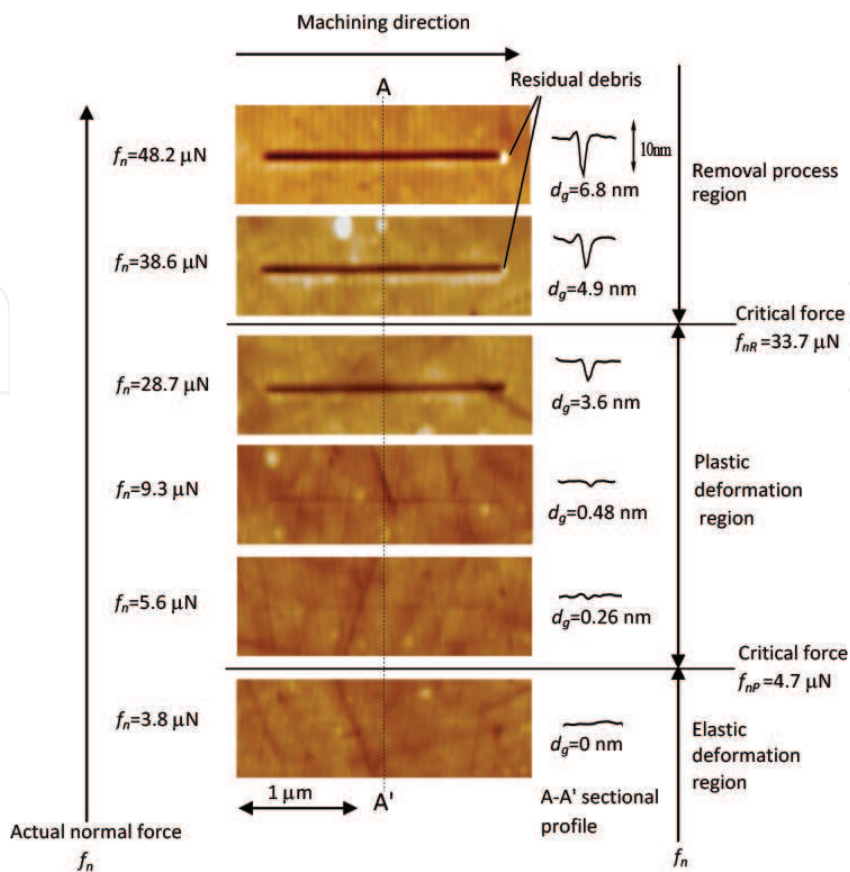
**Table 1.** Machining conditions.

3. Formation of nanometer-scale grooves

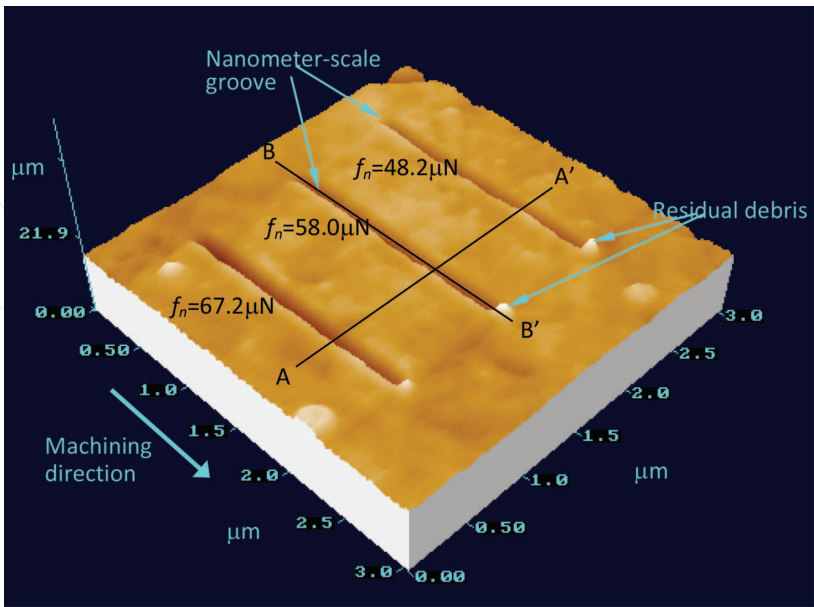
Typical AFM images of the straight grooves with a length of 2  $\mu\text{m}$  processed at different normal forces  $f_n$  of 48.2  $\mu\text{N}$  or less are shown in **Figure 5**. In this figure, the actual normal machining force  $f_n$  measured with the z-axis force transducer, the A-A' sectional profiles of grooves, and the actual mean groove depths  $d_g$  obtained from these profiles are also indicated. Grooves were not formed using normal machining forces of 3.8  $\mu\text{N}$  or less.

But, at a normal force of 5.6  $\mu\text{N}$  a very fine straight groove with a constant depth of about 0.26 nm and a constant width of about 20 nm was successfully generated. From this result, it is confirmed that the minimum normal force needed to form reproducibly a groove on the fused quartz surface is between 3.8 and 5.6  $\mu\text{N}$ . This minimum force is a critical value (denoted by  $f_{nP}$ ) when the plastic deformation begins in this nanomachining. The value of  $f_{nP}$  is given by 4.7  $\mu\text{N}$  that is the average of 5.6 and 3.8  $\mu\text{N}$ .

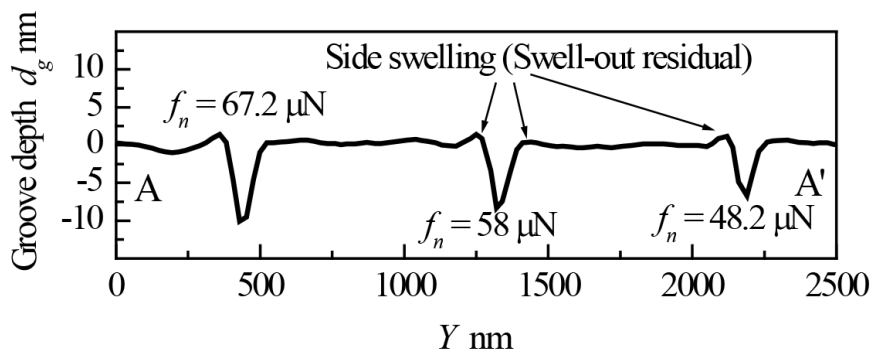
Both depth and width of the straight groove are increased with increasing the normal force. At the same time, small amounts of residual debris can be observed at the end of the groove when the normal force exceeded 28.7 $\mu\text{N}$ . **Figure 6** shows a typical 3D AFM image of the grooves and the residual debris and **Figure 7(a)** and **(b)** show the sectional profiles at sections AA' and BB' in **Figure 6**, respectively. The bottom of the straight groove has a smooth surface with a roughness less than a few nanometers Rz. The side swelling (swell-out residual) is observed at both sides of the straight groove. On the other hand, the protrusion height of the residual debris is much higher than that of the side swelling. Therefore, this residual debris seems to offer evidence that material removal action has occurred.



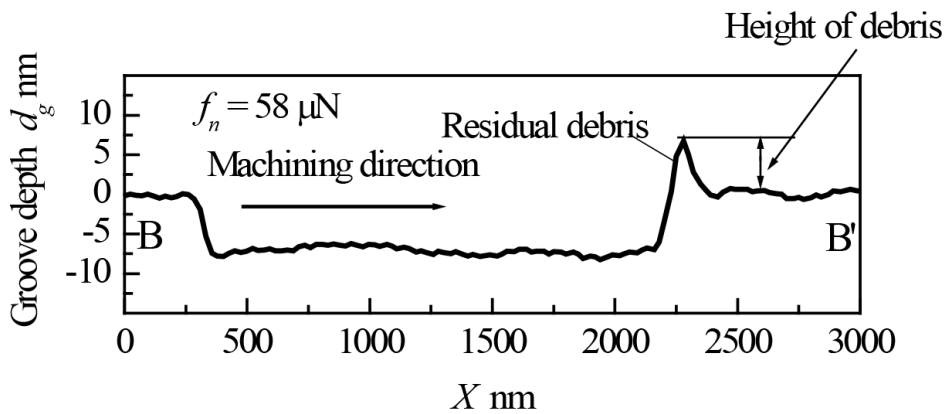
**Figure 5.** Typical AFM images of the straight grooves with a length of 2  $\mu\text{m}$  processed at various normal forces  $f_n$  less than 48.2  $\mu\text{N}$ .



**Figure 6.** AFM image of machined nanometer-scale grooves.



(a) Profile at section AA' in Fig. 6



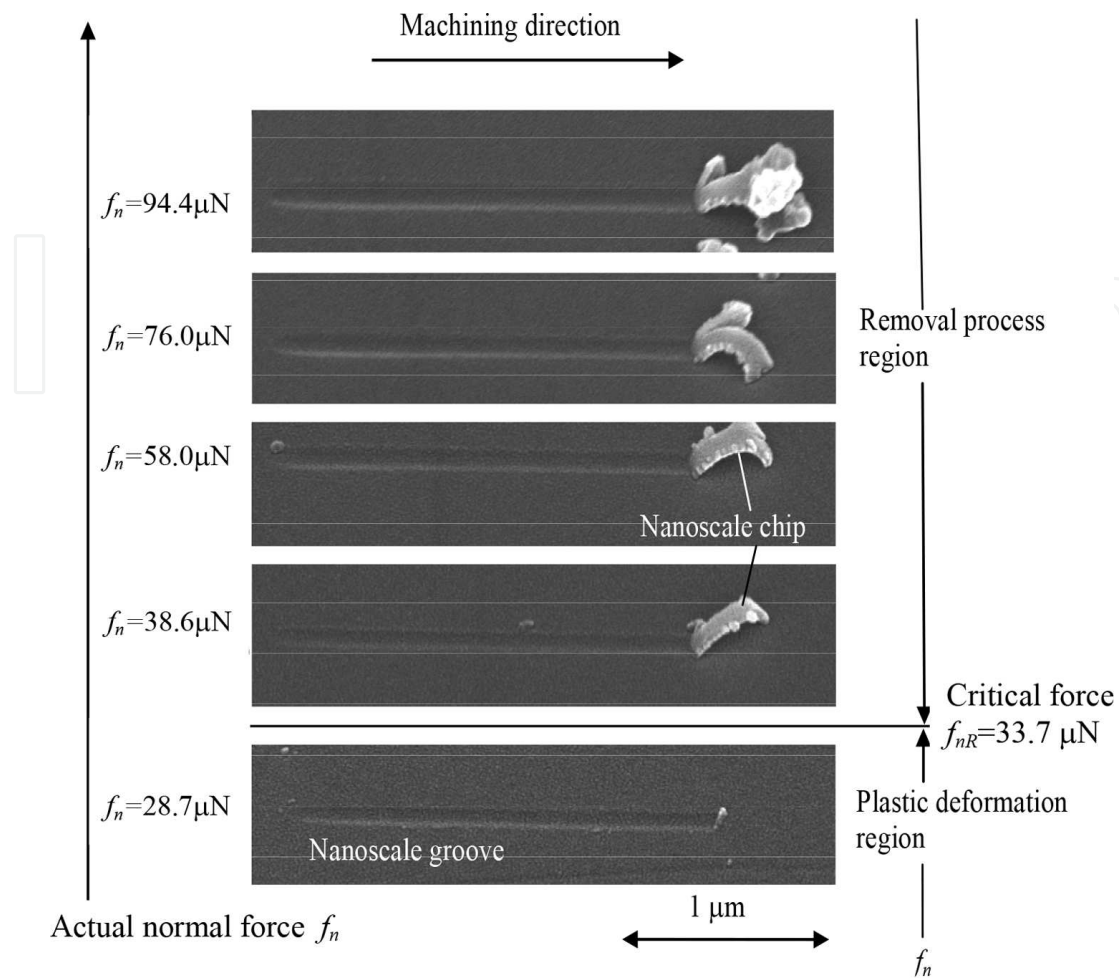
(b) Profile at section BB' in Fig. 6

**Figure 7.** Section profiles of machined nanometer-scale grooves. (a) Profile at section AA' in **Figure 6**. (b) Profile at section BB' in **Figure 6**.

Especially, when the AFM probe scanned the groove surface, the cutting chip that remained at the end of the groove was fractured by the probe. Consequently, such residual debris remained at the end of the groove. Generally, it is easy for the residual cutting chip to be fractured by the probe in scanning for imaging because it projects from the surface. Hence, it is very difficult to observe the cutting chips that remained at the end of the groove after nanomachining, using the AFM.

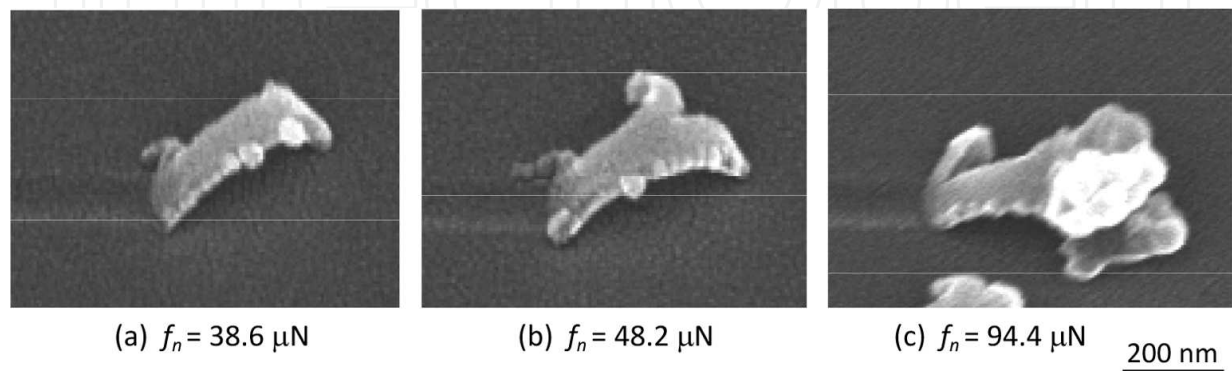
So we tried observing the cutting chips with a field emission scanning electron microscope (FE-SEM/SU8040). **Figure 8** shows the typical observation results. When the normal force was less than  $28.7 \mu\text{N}$ , no cutting chips were observed. But when the normal force was  $38.6 \mu\text{N}$ , a nanometer-scale cutting chip that remained at the end of the groove was grasped. These results show that the minimum normal force when the chip begins to be produced on the fused quartz surface is between  $28.7$  and  $38.6 \mu\text{N}$ .





**Figure 8.** Typical AFM images of the straight grooves with a length of 2  $\mu\text{m}$  processed at various normal forces  $f_n$  of 94.4  $\mu\text{N}$  or less.

**Figure 9** shows the high magnification SEM images of the cutting chips obtained at the normal forces of 38.6, 48.2, and 94.4  $\mu\text{N}$ . Because these cutting chips have a continuous shape and a smooth surface, they can be judged to be a flow-type chip.



**Figure 9.** High magnification SEM images of cutting chips. (a)  $f_n = 38.6 \mu\text{N}$ . (b)  $f_n = 48.2 \mu\text{N}$ . (c)  $f_n = 94.4 \mu\text{N}$ .



These results show that the mechanism of the groove formation, that is, the material removal in nanomachining, is based on the ductile mode minute cutting action that is chiefly performed by the shear deformation. The size of the cutting chip increases with increasing normal force.

These AFM and SEM observations show that the minimum groove depth when the material removal process begins is between 3.6 and 4.9 nm. This minimum groove depth  $d_{gR}$  is an important critical value to indicate the machining unit in this nanomachining process [31], and is given by 4.3 nm as an average of the upper and lower bound values (4.9 and 3.6 nm, respectively). Simultaneously, it is found that the minimum normal force to initiate material removal is between 28.7 and 38.6  $\mu\text{N}$ . The minimum force  $f_{nR}$  is also an important critical value to indicate the machining unit and is given by 33.7  $\mu\text{N}$  (the average of the upper bound value of 38.6 and the lower bound value of 28.7  $\mu\text{N}$ ).

#### 4. Transient behavior from plastic deformation to removal process

The effects of the normal force on the characteristic machining parameters that indicate the features of the groove processed by the diamond edge, that is, the groove depth  $d_g$ , the groove width  $w_g$ , the aspect ratio  $d_g/w_g$  and the swelling ratio  $R_s$  are shown in **Figure 10**. The swelling ratio  $R_s$  is given by  $R_s = (B_1 + B_2)/A$ , where  $B_1$  and  $B_2$  are cross-sectional areas of the swell-out residuals, and  $A$  is a cross-sectional area of the groove, respectively, as shown by the small illustration in **Figure 10**.

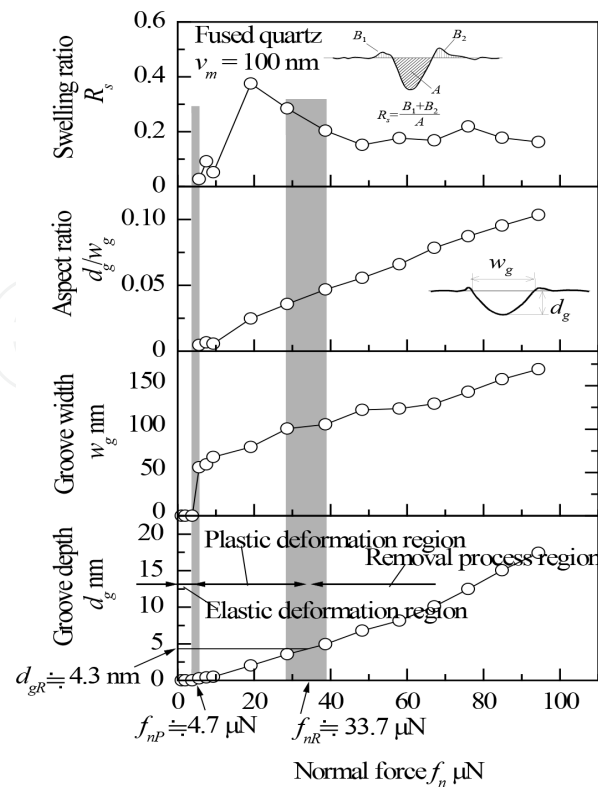
From the above-mentioned results, it is found that the following three regions exist in the process until the fused quartz surface is removed by the diamond tip. These regions are:

- (a)  $0 \leq f_n \leq 4.7 \mu\text{N}$ : Elastic deformation region
- (b)  $4.7 \leq f_n \leq 33.7 \mu\text{N}$ : Elastic/plastic deformation region
- (c)  $33.7 \leq f_n$  ( $d_g \geq 4.3 \text{ nm}$ ): Removal process (cutting process) region

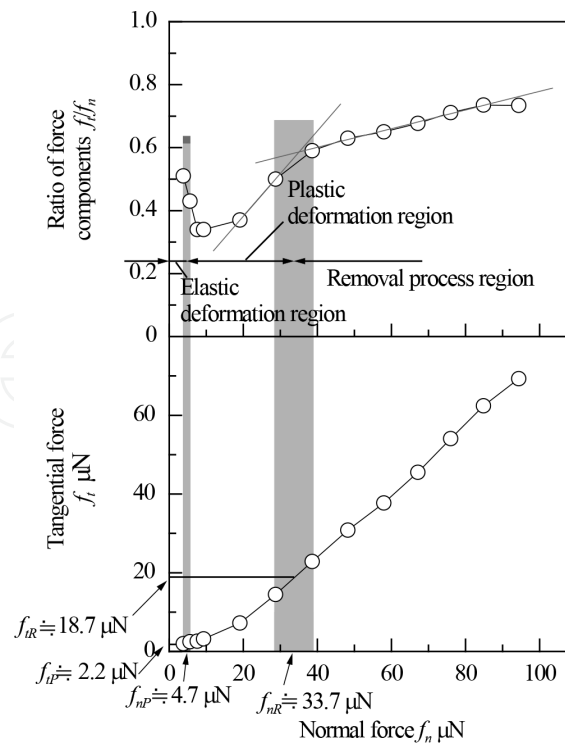
In these expressions, 4.7  $\mu\text{N}$  is the critical normal force  $f_{nP}$  when plastic deformation originates, and its standard deviation is 0.73  $\mu\text{N}$ . On the other hand, 33.7  $\mu\text{N}$  and 4.3 nm are the critical normal force  $f_{nR}$  and groove depth  $d_{gR}$  when removal action originates, and these standard deviations are 4.1  $\mu\text{N}$  and 0.62 nm, respectively.

As shown in **Figure 10**, the swelling ratio  $R_s$  decreases rapidly when the normal force exceeds its critical value  $f_{nR}$ . This feature change in  $R_s$  can be used to identify a transition condition for changing from the plastic deformation to the removal process region.

The changes of tangential force  $f_t$  and ratio of force components with increasing normal force  $f_n$  are shown in **Figure 11**. These results show that the minimum tangential force, namely, the critical value  $f_{tP}$  when the plastic deformation begins, is given by 2.2  $\mu\text{N}$  as an average of the upper and lower bounds (2.46 and 1.96  $\mu\text{N}$ , respectively). Concurrently, the mean minimum tangential force  $f_{tR}$  when the material removal process begins is 18.7  $\mu\text{N}$  (the average of the upper bound/22.8  $\mu\text{N}$  and lower bound/14.5  $\mu\text{N}$ ).



**Figure 10.** Changes of characteristic parameters with increasing normal force  $f_n$ .



**Figure 11.** Effects of normal force on tangential force and ratio of force components.

The main peculiarity in **Figure 11** is a change in the ratio of the force components with increasing the normal force  $f_n$ . Especially, the value of  $f_t/f_n$  changes suddenly from a rapid increase to a gradual increase with increasing the normal force. This feature change in  $f_t/f_n$  is caused by the difference in the direction of the processing force between the plastic deformation and removal process regions [17, 26]. This phenomenon can also be used to identify a critical condition when changing from the plastic deformation to the removal process region.

## 5. Conclusions

Nanometer-scale machining on fused quartz surfaces has been carried out with an AFM combined with a two-axis capacitive force/displacement transducer. The main results obtained in this study are:

- (1) The minimum normal force  $f_{nP}$  required to form reproducibly a groove is about  $4.7\mu\text{N}$ . Namely, the groove is first formed by plastic deformation when the normal force exceeds  $4.7\mu\text{N}$ .
- (2) The minimum critical normal force  $f_{nR}$ , tangential force  $f_{tR}$ , and groove depth  $d_{gR}$  required to initiate the material removal process are, respectively, as follows:

$$f_{nR} = 33.7\mu\text{N}$$

$$f_{tR} = 18.7\mu\text{N}$$

$$d_{gR} = 4.3\text{nm}$$

Namely, the groove is first formed by cutting action when the groove depth exceeds  $4.3\text{nm}$ .

- (3) The characteristic changes in the swelling ratio  $R_s$  and the ratio of force components  $f_t/f_n$  at the critical normal force  $f_{nR}$  can be used to identify a critical condition for changing from the plastic deformation to the material removal process region.
- (4) The mechanism of the material removal in nanomachining is based on the ductile mode minute cutting action that is chiefly performed by the shear deformation.

As described above, the minimum groove depth, that is, the minimum depth of cut when the groove is first removed by the cutting action is a fundamental value called "Machining Unit," which is an important parameter that controls machining accuracy and machining quality [31]. This machining unit will be mainly affected by the tip radius of the diamond tool. To reduce the machining unit, it is necessary to reduce the tip radius of the diamond tool. In the future, we are planning to report on the effects of the tip radius on the machining unit.

## Acknowledgements

This research was supported in part by Grants-in-Aid for General Science Research (C) (No. 19560106) from the Ministry of Education, Culture, Sports, Science and Technology of Japan.

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