Precision in the Mechanical Removal Processing of a Nanosheet by AFM Probe

– Study of the Processing Method for Fabrication of High Precision Grooves –*

Kazuya DEMURA**, Naoki WATANABE**, Yuta OHKI** and Tohru IHARA**

**Department of Precision Mechanics, Chuo University

1-13-27 kasuga, Bunkyo-ku, Tokyo, Japan

E-mail: demura.ihara.lab@gmail.com

Abstract

This paper presents nano mechanical removal processing of a potassium niobate nanosheet with an atomic force microscope (AFM). In particular, we examined the control processing method for the direction of the nanosheet surface in order to attain a high precision groove machining of a bilayer nanosheet. As a result, by the machining direction taking into account machining anisotropy and the machining load W that balance machining accuracy and machining stability, a machined groove with a maximum width of 28nm and a very sharp edge of a bilayer nanosheet was attained.

Key words: Nanosheet, Processing Control, High Precision Groove Machining, Machining Anisotropy, Machining Stability, Machining Accuracy

1. Introduction

This paper presents nanomechanical removal processing of potassium niobate nanosheet using atomic force microscope (AFM). The bilayer nanosheet of potassium niobate is 2 nm thick. The 2 nm of thickness contains a small number of atoms; therefore, the knowledge obtained from experiments done processing a nanosheet is useful when processing properties for the order of atomic size are examined.

Although examples of mechanical removal processing for periodical layered structures such as potassium niobate have been reported using the same processing as done in this paper⁽¹⁾⁻⁽³⁾, the reports were only of control processing of layered direction.

Therefore, in this paper we examine the control processing method for the surface direction of nanosheet, in order to realize a high precision groove machining of a bilayer nanosheet.

2. Explanation of potassium niobate and machining principle

2.1 Potassium niobate nanosheet

Potassium niobate ($K_4Nb_6O_{17} \cdot 3H_2O$) is a periodical layered structure, and its surface has been able to be observed for the atomic size order. The crystal structure of potassium niobate is shown in Fig.1. The nanosheet is formed of two-dimensional layers composed of octahedrons, which consists of niobium (Nb) and oxygen (O) atoms in the ac direction⁽⁴⁾⁽⁵⁾.

In addition, potassium niobate has ion exchange ability⁽⁶⁾. Potassium (K) ions of interlayer1, as shown in Fig.1, are able to exchange organic ammonium ions; thereby, a 2 nm thick bilayer nanosheet is fabricated. These ion exchanges enable us to attain bilayer-level processing.

*Received 9 Feb., 2010 (No. 10-0061)
[DOI: 10.1299/jamdsm.4.858]

Copyright © 2010 by JSME

Figure 1 Crystal structure of potassium niobate

Figure 2 Schematic of nanosheet machining principle

Table 1 Scan mode of processing

Scan mode	Processing type	Measurement of lateral force	Machining load [nN]
Contact	Groove	×	750 ~ 1000
LFM	Pocket	○	100 ~ 415

Table 2 Cantilever type

Scan mode	Cantilever type		
	Material	Force constant [N/m]	Tip radius [nm]
Contact	Silicon	2.1	< 15
		3	< 15
LFM	Silicon nitride	0.05	< 20

2.2 Machining principle

The principle of nanosheet processing is shown in Fig.2: the tip of the AFM probe on the nanosheet is indented by the machining load W , and is scanned by machining speed V .

The processing examined in this paper used the contact mode and lateral force microscope (LFM) mode among various measurement modes of AFM. The control for each mode is shown in Table 1. The contact mode is used for observing the surface and groove machining. The LFM mode is used for measurement of lateral force in machining.

The properties of the cantilever used in each mode are shown in Table 2.

3. Effect of machining anisotropy on nanosheet processing

3.1 Existence of machining anisotropy

Machining anisotropy primarily affects the machining accuracy. Observing the

Figure 3 Surface edge shape of nanosheet

Figure 4 Edge shape of machined circle

nanosheet surface, a surface edge shape of cracked nanosheet consists of cyclic 90 deg angles as shown in Fig.3. Furthermore, the edge shape of a machined circle as shown in Fig.4 was similar to the shape of a cracked nanosheet. This circle machining for a radius of 500 nm was attained with a machining load $W = 500$ nN of contact mode.

We expect that the control of crack propagation improves machining accuracy.

3.2 The direction of machining anisotropy

In the preceding paragraph, machining anisotropy is assumed. Then, the direction of machining anisotropy is derived in this paragraph.

The sheet planes filled with dense atoms are planes $\{1\ 2\ 0\}$, $\{0\ 57\ 37\}$, and $\{3\ 11\ 1\}$. Accordingly, the chemical bonding force F_a of each sheet plane is calculated from an approximation of the Coulomb force for the atoms of the nanosheet. The force F_a ratio is

$$F_a\{1\ 2\ 0\} : F_a\{0\ 57\ 37\} : F_a\{3\ 11\ 1\} = 3 : 8 : 23.$$

The position of three planes on the nanosheet surface is shown in Fig.5. In particular, the positions of planes $\{1\ 2\ 0\}$ and $\{0\ 57\ 37\}$ are at a 90 deg angle on the nanosheet surface. F_a of planes $\{1\ 2\ 0\}$ and $\{0\ 57\ 37\}$ is weak compared to that of plane $\{3\ 11\ 1\}$.

Consequently, the derivation of the bonding force F_a and direction of the sheet plane ensures that the surface edge shape of a cracked nanosheet consists of cyclic 90 deg angles as in Figs.3 and 4.

3.3 Edge stress of planes $\{1\ 2\ 0\}$ and $\{0\ 57\ 37\}$ in groove machining

In groove machining of the direction corresponding to that of plane $\{1\ 2\ 0\}$, the edge stress loaded in plane $\{1\ 2\ 0\}$ and $\{0\ 57\ 37\}$ is calculated using the Hertz contact model as shown in Fig.6. From the model, the direction x and z correspond to the directions of the

Figure 5 Crystal anisotropy of nanosheet surface

Figure 6 Hertz contact model in groove machining

planes $\{0\ 57\ 37\}$ and $\{1\ 2\ 0\}$, respectively. The direction y corresponds to the nanosheet depth.

The contact pressure P is given by

$$P = P_m \sqrt{1 - \left(\frac{x}{a}\right)^2}, \quad (1)$$

$$P_m = \frac{3F}{2A}, \quad (2)$$

$$a \cong \left[\frac{3}{4} R \left(\frac{1 - \nu_1^2}{E_1} + \frac{1 - \nu_2^2}{E_2} \right) F \right]^{1/3}, \quad (3)$$

where F and A are machining force and area of cross section, respectively of a nanosheet with contact width $2a$ and thickness 2 nm . E_1 and ν_1 and E_2 and ν_2 are Young's modulus E and Poisson's ratio ν of the nanosheet and silicon probe, respectively.

The maximum tensile stress at the contact edge when $x = a$ is given by

$$\sigma_m = \frac{1 - 2\nu}{3} P_m. \quad (4)$$

The stress tensor $\mathbf{B}$ taking account of surface friction between the silicon probe's tip and the nanosheet at the contact edge is given by

$$\mathbf{B} = \begin{pmatrix} \sigma_x & \tau_{xy} & \tau_{zx} \\ \tau_{xy} & \sigma_y & \tau_{yz} \\ \tau_{zx} & \tau_{yz} & \sigma_z \end{pmatrix} = P_m \begin{pmatrix} \frac{1 - 2\nu}{3} & 0 & \mu \\ 0 & 0 & 0 \\ \mu & 0 & 0 \end{pmatrix}, \quad (5)$$

Figure 7 Relationship between machining force F and tensile stress of slipping plane

Figure 8 Machined groove direction is oblique 45 deg from the direction of plane $\{1\ 2\ 0\}$

where $\sigma_y = 0$ because the stress in the direction y of the nanosheet depth suppose not to affect the machining of the nanosheet surface.

The edge tensile stress σ of the planes $\{1\ 2\ 0\}$ and $\{0\ 57\ 37\}$, which is calculated by Eqs. (1) ~ (5), where $F = 200 \sim 600$ nN, $R = 15$ nm, $E_1 = 8$ GPa, $E_2 = 190$ GPa, $\nu = \nu_1 = \nu_2 = 0.3$, $\mu = 0.6$, is shown in Fig.7. The values of E_1 and μ were derived from experiments. The tensile stress of plane $\{1\ 2\ 0\}$ is equal to that of plane $\{0\ 57\ 37\}$. By contrast, the bonding force F_a $\{0\ 57\ 37\}$ calculated in the preceding paragraph is about 2.7 times as large as that of plane $\{1\ 2\ 0\}$. Consequently, the direction generating a crack first is expected to be the direction of the $\{1\ 2\ 0\}$ plane.

The machined groove in the machining direction is oblique at 45 deg angle based on the direction of plane $\{1\ 2\ 0\}$ and is shown in Fig.8. The cracked edge of plane $\{1\ 2\ 0\}$ is larger than that of plane $\{0\ 57\ 37\}$.

In the next paragraph, we examine the machining accuracy in the direction of plane $\{1\ 2\ 0\}$.

3.4 Groove machining taking into account machining anisotropy

The machined groove in the direction corresponding to that of plane $\{1\ 2\ 0\}$ is shown in Fig.9 (a), and the cross section is shown in Fig.9 (b). This groove was machined with a maximum width around 34 nm, and a very sharp edge is attained.

Consequently, using machining anisotropy one can attain a high precision and efficient groove machining which is controlled as the crack propagates in the direction of plane $\{1\ 2\ 0\}$. Next, machining is performed in the direction corresponding to that of plane $\{1\ 2\ 0\}$.

4. Machining stability and accuracy

4.1 Machining stability distinguished the periodicity of machining force

There was a small difference in width in the same machined groove. The reason is assumed to be the effect of the machining force. Accordingly, the machining force is measured with LFM.

(a) Machined groove direction of plane $\{1\ 2\ 0\}$

(b) Cross section of machined groove

Figure 9 Machined groove taking into account machining anisotropy

Figure 10 Relationship between machining load W and the average of machining force F

With a machining speed $V = 200$ nm/s and machining load $W = 70 \sim 800$ nN, the machining force F is measured against a bilayer nanosheet. The relationship between machining load W and the average machining force F is shown in Fig.10. The average machining force F tends to be proportional to the machining load W .

The frequency of the machining force is analyzed by FFT. The result is shown in Fig.11. The frequency is determined by the second peak. Because the first peak $1 \sim 5$ Hz is observed when no machining is being done; this peak is identified as the eigen frequency of the measurement system. Periodicity distance of machining force calculated from the second peak of frequency is shown in Fig.12. This distance tends to be proportional to machining load W .

If the periodicity distance of the machining force is short, the machining is expected to be intermittent and defined as unstable. On the other hand, when the distance is long, the machining is defined as stable.

Figure 11 Relationship between frequency and power

Figure 12 Relationship between machining load W and periodicity distance of machining force

4.2 Machining stability distinguished the periodicity of machining force

The machining load W is needed for machining stability. In this paragraph, we examine the relationship between machining load W and groove machining accuracy.

The groove machining accuracy is evaluated from the machined groove coarseness Δw . Δw is given by

$$\Delta w = w_{\max} - w_{\min} \quad (6)$$

The evaluation of $w_{\max}$ and $w_{\min}$ in Eq. (6) is as shown in Fig.13. When Δw is small, precision groove machining is attained.

Groove machining of a bilayer nanosheet is performed with a machining speed $V = 200$ nm/s and a machining distance $2 \mu\text{m}$. The relationship between machining load W and groove machined coarseness Δw is shown in Fig.14. Δw tended to be proportional to machining load W .

Machining accuracy tends to be inversely proportional to machining stability. Therefore, machining load W must be balanced with machining accuracy and machining stability so that high precision groove machining is attained.

5. Realization of a high precision groove machining

The point $\Delta w = 0$ in Fig.14 is identified as the lower limit of machined groove width w_0 attained with high precision groove machining. The point (W_0, w_0) in Fig.14 is calculated as (650, 25) owing to the linear functions of $w_{\max}$ and $w_{\min}$ using the least squares method.

Figure 13 Evaluation of minimum and maximum machined groove width

Figure 14 Relationship between machining load W and machined groove width w

However, high precision groove machining is not always attained with the machining load of $W = 650$ nN, if machining stability is not considered. Accordingly, precision groove machining is performed with a machining speed $V = 200$ nm/s and the machining load $W = 800$ nN that balances machining accuracy and machining stability. The machined groove is shown in Fig.15. The edge of the machined groove as shown in Fig.15 (a) is very sharp. Furthermore, $w_{\max}$ as shown in Fig.15 (b) is about 28 nm, which is nearly equal to $w_0 = 25$ nm.

The processing method controlled machining load W taking into account machining anisotropy and machining stability helps attain a high precision groove machining of a bilayer nanosheet for the surface direction.

6. Conclusion

In order to control the processing of a bilayer nanosheet for surface direction, it is important to take into account machining anisotropy.

In addition, the machining stability proved to be proportional to machining load W from the periodicity distance of machining force. In contrast, the machining accuracy proved to be inversely proportional to machining load W from machined groove coarseness Δw . Therefore, machining load W must balances with machining stability and machining accuracy.

By the machining direction taking into account machining anisotropy and the machining load W that balances machining stability and machining accuracy, a machined groove with a maximum width of 28 nm and a very sharp edge of bilayer nanosheet was attained.

(a) High precision machined groove

(b) Cross section of high precision machined groove

Figure 15 Machined groove taking into account machining anisotropy and machining stability

References

- (1) Miyake, S., Ishii, M., Otake, T. and Tsushima, N., 1997, Nanometer-scale Mechanical Processing of Muscovite Mica by Atomic Force Microscope, *Journal of the Japan Society of Precision Engineering*, Vol.63, No.3, pp.426-430(in Japanese).
- (2) Miyake, S., Otake, T. and Asano, M., 1999, Mechanical Processing Mica Using an Atomic Force Microscope, *Journal of the Japan Society of Precision Engineering*, Vol.65, No.4, pp.570-574(in Japanese).
- (3) Miyake, S., Sekine, Y., Kim, J., Yamamoto, H., 2000, Development of Nanofabrication Technique Based on Wear Properties of Nanoperiod Multilayerd Films, *Journal of the Japan Society of Precision Engineering*, Vol.66, No.12, pp.1958-1962(in Japanese).
- (4) H. Shindo, M. Kaise, H. Kondoh, C. Nishihara, H. Hayakawa, S. Ono, H. Nozoye, Structure of the cleaved surface of potassium niobate hydrate ($K_4Nb_6O_{17} \cdot 3H_2O$) studied by atomic force microscopy, *Langmuir*, Vol. 8 (1992) pp. 353-356.
- (5) H. Shindo, H. Nozoye, Cleavage and Intercalation Properties of a Layered Compound, $K_4Nb_6O_{17} \cdot 3H_2O$, Studied with Atomic Force Microscopy, *Japanese Journal of Applied Physics*, Vol.32 (1993) pp.2950-2951.
- (6) N. Miyamoto, H. Yamamoto, R. Kaito and K. Kuroda, Formation of extraordinarily large nanosheets from $K_4Nb_6O_{17}$ crystals, *Chemical Communications*, 2002, pp.2378-2379.