Study of ultrasound-assisted nanomachining on monocrystalline silicon

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Abstract. This study constructed an ultrasound-assisted AFM nanomachining platform by integrating an atomic force microscope (AFM) with a quartz crystal microbalance, which was subsequently used in experiments for determining the microscopic phenomena of ultrasound-assisted nanomachining. Force–distance curve measurement was used to establish a normal force measurement model, obtaining the downforce strengths during ultrasound-assisted nanomachining. Subsequently, machining experiments were conducted on monocrystalline silicon to explore the effects of various experimental parameters (e.g., machining speed and normal force) on machining depth. The experiments showed that the ultrasound-assisted AFM nanomachining platform constructed in this study is effective. After ultrasound was incorporated, monocrystalline silicon specimens made of brittle material could be more effectively processed under various experimental parameters. Moreover, the performance of ultrasound-assisted nanomachining was superior to that of general nanomachining.

1. Introduction

The atomic force microscope (AFM) was invented by Binnig et al. [1] in 1986. It is an improved version of the scanning tunneling microscope, which is limited by its requirement of conductive materials. The AFM overcomes the shortfall of the scanning tunneling microscope in that research materials are no longer confined to conductive materials. AFMs can be used to measure the morphology of conductor surfaces and examine the surface morphology of nonconductive materials. The AFM not only yields images with high spatial resolution but it can also operate in a vacuum, making it an indispensable instrument in the development of nanotechnologies.

The applications of AFM probes in nanomachining have been topic of research in recent years. For example, in 1989, Loenen et al. [2] proposed using a tungsten probe in an AFM to carve a silicon surface directly. The proposal of this pioneering idea gave rise to the notion of using atomic probes to explore tribological prototypes. Subsequently, Burnham et al. [3] investigated surface forces by probing thin layers with an AFM. Hamada and Kaneko [4–6] have employed atomic probes to analyze microwear and friction forces on the surface of macromolecular materials. Since then, AFMs have been widely used in tribological analysis. In 1997, Woodland and Unertl [7] examined the initial wear of polystyrene surfaces in contact with the probe tips and determined the relationship between downforce and maximum stress based on the deflection of a measuring probe tip. They found that polystyrene films with high molecular weight were more resistant to wear than films with low...
molecular weight. Khurshudov and Kato [8] examined the microwear of polycarbonate using line-scratching and multiple scratching with a Si3N4 AFM tip with radius of 10–20 nm. Bhushan [9] applied AFM diamond tips to create a nanogroove on a silicon substrate. As shown in Fig. 5, no chips were found around the edge of the groove, because the silicon substrate was hard and brittle.

In 2011, Huang [10,11] explored the scratches on CrN–Cu multilayer thin films and related machining behaviors in air and liquid environments using an AFM to conduct nanoscale testing on the surface of these thin films. Huang analyzed the depth and friction forces of machining to evaluate the effects of various machining parameters (e.g., initial condition of tip, normal force strength, machining frequency and speed, and thickness in bilayer period) on the nanomachining behavior of multilayer thin films.

In 2011, Huang [12] employed AFM to examine the nanomachining properties and nanopatterning capabilities of a material. Using mica, polycarbonate, gold, and other materials, Huang conducted linear nanomachining under different downforce conditions to elucidate the scratch patterns, chips, and nanopatterning capabilities of differently processed materials. This study also successfully produced nanopatterns of circles with a diameter of 500 nm and straight lines on gold specimens.

In the 70s and 80s, ultrasound machining was widely applied in abrasive flow machining, grinding, plastic deformation processing, welding, soldering, metallurgy, and ultrasonic cleaning. It is currently adopted for precision cutting and ultraprecision machining. In ultrasonic vibration cutting, both cutting motion and relative oscillation play a role in achieving the ideal cutting effect between a cutting tool and workpiece. When a vibration source is placed in between the cutting tool and workpiece, a tiny gap is instantaneously generated between the cutting tool and the chips, forming a near-vacuum state. A layer of oxide film formed on the contact surface between the cutting tool and the chips can decrease interfacial frictions and subsequently reduce cutting force, thereby improving the roughness of the cutting surface.

Numerous scholars have therefore incorporated ultrasonic vibration in micromachining processes to reduce cutting force and improve the quality of machining works. Ultrasound-assisted processes integrated with an AFM are used mostly for measurement. For example, acoustic atomic force microscopy (AFAM) [13] and ultrasonic friction force microscopy (UFFM) [14,15] are employed to detect resonance phenomena between a probe and workpiece.

Scholars have also used AFM to conduct ultrasound-assisted nanomanipulation. In 2010, Lytvyn et al. [16] grew a 100-nm-thick layer of native germanium nanoparticles on a silicon wafer and conducted nanomanipulation of the germanium nanoparticles using an AFM probe combined with ultrasonic vibration.

Despite the aforementioned discussion, the construction and investigation of ultrasound-assisted nanomachining experimentation systems and simulations are scant in international publications. Because the scale and depth of nanomachining are small, the cutting force resulting from a machining process and the postmachining surface quality are closely related to the integrity and functions of finished products. Therefore, ultrasound-assisted nanomachining warrants further investigation. This study provides a comprehensive overview of the numerous phenomena and properties of ultrasound-assisted nanomachining with an AFM as reference for relevant practitioners.

2. Experimental Planning and Methods

2.1. Specimen Preparation
A nanoindenter was used to analyze the hardness and Young’s modulus of a monocrystalline silicon specimen and ascertain whether the specimen was brittle. The hardness of six areas of the specimen was measured to obtain an average. An indentation curve is presented in Fig. 1, and the measured values are summarized in Table 1. The results show that the average hardness of the specimen was 10.52 GPa, and the elasticity coefficient was 167.8 GPa; these measurements indicate that the specimen was brittle.
Figure 1. Indentation curve of monocrystalline silicon specimen

Table 1. Average nanoindentation hardness value of monocrystalline silicon specimen.

| Er(GPa) | Hardness(GPa) | Contact Depth(nm) | Max Depth(nm) |
|--------|--------------|-------------------|--------------|
| 167.8  | 10.52        | 60.25             | 79           |

Subsequently, a silicon wafer was cut into appropriate sizes. The specimen used in this experiment weighed approximately 0.086 g. The specimen was immersed in isopropyl alcohol, placed in an ultrasonic cleaner for 5 min, wiped with absolute alcohol (99.5%), and finally air-dried with a dust blower.

Fast-acting adhesive was used to attach the silicon specimen to a quartz crystal microbalance (QCM) resonator, as shown in Fig. 2. The specimen was then tested on the QCM. The ultrasonic resonance frequency was an average of 5.0 MHz, and resistivity was approximately 162 Ω, suggesting that ultrasonic vibration was feasible.

2.2. Probe Preparation
This study adopted a diamond-coated probe to conduct nanomachining experiments. A DCP-20 probe (NT-MDT, Russia) was selected. The specifications and scanning electron microscope (SEM) images of this probe are shown in Table 2 and Fig. 3, respectively.

A diamond-coated probe was used to conduct a nanomachining experiment. To obtain the probe’s elasticity coefficient (Kn) precisely, the real resonance frequency (f) of the probe must be measured. The elasticity coefficient is proportional to the square of resonance frequency [23], expressed as follows:

\[
\frac{f_0}{f} = \frac{\sqrt{K_n}}{\sqrt{K}}
\]

(1)

where \(f_0\) and \(K_n\) represent the resonance frequency and elasticity coefficient of the probe provided by the original manufacturer, \(f\) represents the measured resonance frequency of the probe, and \(K\) represents the actual calculated elasticity coefficient of the probe. The shape and size of the probe can be obtained using an electronic microscope.
Table 2. Specification of DCP-20 probe.

| Specification                                      | Value                                      |
|----------------------------------------------------|--------------------------------------------|
| Cantilever thickness /Length (L)/Width (W)         | 1.7-2.3 μm / 90 μm / 60 μm                 |
| Resonance frequency                                | 260-630 KHz                                |
| Force constant                                     | 48 N/m                                     |
| Tip height                                         | 10-15 μm                                   |
| Tip coating                                        | Diamond coating                            |

2.3. Force–Distance Curve

A force–distance (f–d) curve method involves using a probe to compress and decompress specimen vertically. Fig. 4 displays the displacement of the probe’s deflection along the Z-axis; when the probe contacts the specimen, it follows the sequence A→B→C→D shown in the figure, and the order D→C→B→A results when the probe moves away from the specimen. The scan setpoint is point D in Fig. 4. Therefore, the extent to which the probe deflects in the vertical direction is the distance d between points D and F. The slope at points D and F is referred to as the normal sensitivity Sz of the deflection in the vertical direction.

Normal force ($F_n$) is equivalent to the actual downforce between a probe and specimen and is calculated by multiplying distance $d$ by the positive elasticity coefficient $K_n$ of the probe. In other words, $F_n = K_n d$.

3. Conclusion Construction of Ultrasound-Assisted AFM Nanomachining Platform and Experimental Planning

3.1. Construction of Ultrasound-Assisted AFM Nanomachining Platform

The ultrasound-assisted AFM nanomachining platform was constructed by integrating a D3100 AFM (Veeco, USA) (Fig. 5), QCM200 (Stanford research systems, USA) (Fig. 6), and a crystal holder (Fig. 7). The complete setup is shown in Fig. 8. The platform generates a resonance frequency of 5 MHz and oscillates vertically.

Figure 4. The f–d curve

Figure 5. D3100 AFM

Figure 6. QCM200
3.2. Experimentation
This study conducted experiments to investigate the effects of machining speed, downforce (normal force), number of repeats, and other parameters on the depth of machining. The constructed nanomachining platform generated an ultrasound resonant frequency of 5 MHz and oscillated up and down. The probe initially moved at 0° to scan the surface and subsequently at 90° during the process while applying load, as illustrated in Fig. 9. The scope of machining was set at 5 × 5 μm. The probe moved at either 3, 5, or 7 μm/s coupled with 25–32 μN of force and either ten or five rounds (t) of machining. Subsequently, nanomachining experiments were conducted. The resonance frequency measured by the probe was 474.13 KHz. The actual force constant calculated was 61.1 N/m. Table 3 summarizes the experiments in this study.

4. Results and Discussion
4.1. Ultrasound-Assisted AFM Nanomachining Platform Testing
Samples were subject to ultrasound-assisted measurements using a scan range of 5 × 5 μm. Fig. 10 shows AFM images of the silicon surface. Samples were scanned after ultrasonic vibration was
activated. The scan images were slightly fuzzy, but the platform could still perform measurements as usual. The test results indicated that the ultrasound-assisted AFM nanomachining platform could function normally.

![Figure 10](image)

**Figure 10.** AFM images of silicon surface scanned with (a) ultrasound and (b) without ultrasound

### 4.2. Downforce Effect

The probe was set to exert a downward force of 25–32 μN, moving at 5 μm/s for ten rounds of machining. The results are shown in Figs. 11–13 and Table 4.

Figs. 11–13 reveal that no chips were accumulated on the two sides of the processed area after machining. This study asserts that because monocrystalline silicon is brittle, the process of machining is likely to leave a few brittle chips. However, because ultrasonic vibration was introduced, the probe removed these tiny chips as it scanned the surface.

According to Table 4, ultrasound-assisted AFM nanomachining clearly cut deeper than did general nanomachining. Additionally, the greater the downforce applied, the more prominent was the effect of ultrasonic assistance.

![Figure 11](image)

**Figure 11.** Ten rounds of (A) ultrasound-assisted machining and (B) general machining of monocrystalline silicon at 5 μm/s and 25 μN

![Figure 12](image)

**Figure 12.** Ten rounds of (A) ultrasound-assisted machining and (B) general machining of monocrystalline silicon at 5 μm/s and 27 μN
Figure 13. Ten rounds of (A) ultrasound-assisted machining and (B) general machining of monocrystalline silicon at 5 µm/s and 32 µN

Table 4. Average Cutting Depth of silicon under different downforce strength (unit: nm).

| Force (µN) | Ultrasound Machining | General Machining |
|------------|----------------------|-------------------|
| 25         | 13.9                 | 9.1               |
| 32         | 28.4                 | 36.9              |

4.3. **Probe Speed Effect**

The effect of the probe movement speed was investigated by applying a downforce of 27 µN during ten rounds of machining. Experiments were conducted at 3 and 7 µm/s. The results are provided in Figs. 14 and 15 and Table 5.

Table 5 indicates that at 7 µm/s, ultrasound-assisted machining cut 28 nm deep, whereas general machining cut 17.4 nm deep. This study also found that during ultrasound-assisted machining, the cut was deeper at the high probe moving speed than at low moving speed possibly because a fast-moving cutting tool (probe) exerts a great impact on a hard material workpiece, which facilitates cutting. Thus, ultrasound-assisted machining at high speed produces a more favorable cutting effect than that at low speed.

Figure 14. Ten rounds of (A) ultrasound-assisted machining and (B) general machining of monocrystalline silicon at 3 µm/s and 27 µN

Figure 15. Ten rounds of (A) ultrasound-assisted machining and (B) general machining of monocrystalline silicon at 7 µm/s and 27 µN

Table 5. Average Cutting speed of silicon under different downforce strength (unit: nm).

| Speed (µm/s) | Ultrasound Machining | General Machining |
|--------------|----------------------|-------------------|
| 3            | 18.8                 | 17.3              |
| 7            | 28.0                 | 17.4              |
5. Conclusion
This study constructed an ultrasound-assisted AFM nanomachining platform by integrating an AFM with a QCM; F–d curve measurement was used to establish a normal force measurement model, obtaining the downforce strengths during ultrasound-assisted nanomachining. Subsequently, machining experiments were conducted on monocrystalline silicon to explore the effects that different experimental parameters (e.g., machining speed, normal force, and machining frequency) had on machining depth. The experiments showed that the ultrasound-assisted AFM nanomachining platform constructed in this study is effective.

Ultrasound-assisted nanomachining cut deeper than did general nanomachining. Additionally, greater downforce applied created a more prominent effect of ultrasonic assistance. During ultrasound machining, a deeper cut was created at a high probe moving speed (7 μm/s) than at a low moving speed. The number of rounds more strongly influenced the average cutting depth of ultrasound-assisted nanomachining than that of general machining.

References
[1] G. Binnig, H. Rohrer, Ch. Gerber, E. Weibel, Phys. Rev. Lett. 49 57-61 (1982).
[2] E.J. Von Loenon, D. Dijkkamp, A.J.Hoeven, J. M. Lenssinck,and J. Dieleman, “Direct writing in Si with a scanning tunneling microscope,” Appl. Phys. Lett., Vol.55, pp.1312-1320 (1989).
[3] N. A. Burnham, D. D. Domingues, “Probing the surface forces of monolayer films with an atomic-force microscope,” Phys. Rev.Letters, Vol.64, pp.1931-1934 (1990).
[4] E. Hamada and R. Kaneko, “Microdistortion of polymer surface by friction,” J. Phys. D.: Appl. Phys. A., Vol.53-56, pp.31-34 (1992).
[5] E. Hamada and R. Kaneko, “Micro-tribological evaluation of a polymer surface by atomic force microscopes”, Ultramicroscopy, Vol.42-44, pp.184-190 (1992).
[6] R. Kaneko, and E. Hamada, “Microwear process of polymer surface,” Wear, 162-164, pp.370-377 (1993).
[7] D. D. Woodland, and W. N. Unertl, “Initial wear in nanometer-scale contacts on polystyrene,” Wear, Vol.203-204, pp.685-691 (1997).
[8] A. Khurshudov and K Kato, “Wear mechanisms in reciprocal scratching of polycarbonate, studied by atomic force microscopy,” Wear, Vol.205, pp.1-10 (1997).
[9] Bharat Bhushan, Nano- to microscale wear and mechanical characterization using scanning probe microscopy, Wear ,251, 1105–1123 (1997).
[10] Jen-Ching Huang, Jyh-Wei Lee, Chia-Lin Li, 2011, “Nano-scratching and nano-machining in different environments on Cr2N/Cu multilayer thin films”, Thin Solid Films, Vol. 519 pp.4992–4996.
[11] J.-C. Huang, C.-L. Li, J.-W. Lee, 2011, “The study of nanoscratch and nanomachining on hard multilayer thin films using atomic force microscopy”, Scanning (accepted).
[12] Jen-Ching Huang, Yung-Jin Weng, 2011, “A study of the nanomachining and nanopatterning on Different Materials Using Atomic Force Microscopy”, 2012 AMEE Workshop on Nanoelectronics and Optoelectronics.
[13] Rabe U, Arnold W (1994) Acoustic microscopy by atomic force microscopy. Appl Phys Lett 64:1493.
[14] Scherer V, Arnold W, Bhushan B (1999) Lateral force microscopy using acoustic force microscopy. Surf Interface Anal 27:578.
[15] Reinstein M, Rabe U, Scherer V, Turner JA, ArnoldW(2003) Imaging of flexural and torsional resonance modes of atomic force microscope cantilevers using optical interferometry. Surf Sci 532:1152.
[16] P.M. Lytvyn, O.Ya. Olikh, O.S. Lytvyn1, O.M. Dyachyns’ka, I.V. Prokopenko," Ultrasonic assisted nanomanipulations with atomic force microscope",Semiconductor Physics, Quantum Electronics & Optoelectronics, V. 13, N 1. (2010),PP. 36-42