Study on the vertical ultrasonic vibration-assisted nanomachining process on single-crystal silicon

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Abstract

Subsurface damage that is caused by mechanical machining is a major impediment to the widespread use of hard–brittle materials. Ultrasonic vibration-assisted macro- or micromachining could facilitate shallow subsurface damage compared with conventional machining. However, the subsurface damage that was induced by ultrasonic vibration-assisted nanomachining on hard–brittle silicon crystal has not yet been thoroughly investigated. In this study, we used a tip-based ultrasonic vibration-assisted nanoscratch approach to machine nanochannels on single-crystal silicon, to investigate the subsurface damage mechanism of the hard–brittle material during ductile-machining. The material removal state, morphology, and dimensions of the
nanochannel, and the effect of subsurface damage on the scratch outcomes were studied. The materials were expelled in rubbing, plowing, and cutting mode in sequence with an increasing applied normal load and the silicon was significantly harder than the pristine material after plastic deformation. Transmission electron microscope analysis of the subsurface demonstrated that ultrasonic vibration-assisted nanoscratching led to larger subsurface damage compared with static scratching. The transmission electron microscopy results agreed with the Raman spectroscopy and molecular dynamic simulation. Our findings are important for instructing ultrasonic vibration-assisted machining of hard–brittle materials at the nanoscale level.

**Keywords:** vibration-assisted nanoscratch, single-crystal silicon, subsurface damage

1. **Introduction**

Increased focus has been given to the use of hard–brittle materials, such as single-crystal silicon, owing to their excellent physical and mechanical properties [1,2]. Nanostructures on silicon-based materials are used extensively in many fields [3,4]. Several approaches have been used to fabricate micro- and nanostructures on silicon, such as reactive-ion etching, focused ion-beam lithography, and electron-beam lithography [5-7]. However, these methods are limited by disadvantages, such as a high facility cost, strict environmental requirements, and a low processing efficiency.

Mechanical machining is a more powerful and feasible method to machine single-crystal silicon [8,9]. However, subsurface damage results because of the tool–workpiece contact during machining, which affects the mechanical, electronic, and optical performance of silicon-based devices. Ultrasonic vibration-assisted machining
(UVM) is used extensively to suppress the propagation of subsurface damage. UVM facilitates shallow subsurface damage compared with conventional machining [10-14]. Many researchers have reported that UVM improves the critical depth of the brittle–ductile transition, reduces the scratch load, and inhibits microcrack propagation [15,16]. However, the depth of material removal for the UVM process mentioned above was at the macro- or microscale. To the best of the authors’ knowledge, UVM with a nanoscale machined depth has not been conducted on hard–brittle materials (such as silicon), and the subsurface damage mechanism that caused by nanoscale-UVM has not been investigated.

Atomic force microscopy (AFM) tip-based nanoscratching has been proven as a powerful technique to machine structures with a nanoscale depth of material removal [17,18]. Thus, this technique is suitable for investigating the subsurface damage mechanism of single-crystal silicon at the nanoscale. Lee [19] conducted conventional tip-based nanoscratching on silicon. The machining characteristics and brittle–ductile transitions were monitored by using acoustic emission approach. However, the subsurface damage that was caused by nanoscratching was not studied. Qian et al. [20] achieved the nondestructive fabrication of nanochannels on silicon using a tip-based tribochemistry induced direct nanomachining approach. Wang et al. [21] investigated the subsurface damage of single-crystal silicon that was induced by tip-based nanomilling. They revealed the subsurface, including many dislocations, stacking faults and a layer of amorphous silicon. Nevertheless, the nanomilling was close to conventional tip-based nanomachining rather than ultrasonic vibration-assisted
nanomachining. A vertical Z-axis vibration-assisted tip-based machining approach was carried out by Park et al. [22] to fabricate nanochannels on a gold layer. The method could increase the groove depth significantly. Dong et al. [23,24] fabricated three-dimensional nanostructures on polymethyl methacrylate samples with the aid of ultrasonic tip-sample Z-vibration. A tip-based scratching method with vertical Z-axis vibration was used by Geng et al. [25] to machine the polymer resist. A deeper machined depth and less tip wear were observed compared with the conventional machining technique. However, in contrast with polymers, single-crystal silicon was brittle with little ductility during machining, which makes it difficult to machine. Tip-based ultrasonic vibration-assisted nanoscratching (UVS) that is conducted directly on silicon has not been investigated, and the induced subsurface damage is unclear. Thus, the machining characteristics (i.e., the mechanism of material removal and subsurface damage) should be thoroughly understood for using UVS on single-crystal silicon.

The objective of this study was to investigate nanochannel machining on single-crystal silicon using a tip-based UVS approach. This paper is structured as follows. Section 2 describes the experimental details, and includes the machining setup and molecular-dynamics (MD) simulation model. Section 3 analyzes and compares the machining outcomes (i.e., the mechanism of material removal and subsurface damage) of nanochannels that were fabricated by using UVS and static scratching, respectively. By using a double-pass scratch approach, the effect of subsurface damage on the machining mechanism was investigated. The main conclusions are given in Section 4. Our findings are significant for understanding the mechanism of subsurface damage.
that is caused by ultrasonic vibration-assisted nanomachining on hard–brittle material.

2. Experimental details

2.1 Setup and methods

A commercial AFM with a Nanoscope-V controller (Dimension Icon, Bruker Company, Germany) was used to conduct the fabrication experiments. Figure 1(a) shows a schematic diagram of the AFM-based UVS setup. The sample was attached to a piezoelectric actuator (OD12-F2MHz, Core Tomorrow Science & Technology Co., Ltd., China) with the aid of vacuum grease to avoid vibration energy dissipation. The piezoelectric actuator achieved a megahertz-level vibration in the vertical Z-axis direction under the action of a driving signal that was generated from a commercial signal generator (AFG1022, Tektronix, USA). Figure 1(b) and (c) provides scanning electron microscopy (SEM) micrographs and schematic diagrams of the triangular pyramidal diamond tip (PDNISP, Bruker, Germany) that conducted the machining. The geometric angles of the tip, such as a side ($\theta_s$), back ($\theta_b$), and front ($\theta_f$) angle, as shown in Figure 1(d) and (e), are 51°, 35°, and 55°, respectively. The normal spring constant ($K_N$) and resonance frequency of the tip cantilever that were provided by the manufacturer were 286 N/m and 69.2 KHz, respectively. The tip radius ($R$) was $\sim$86 nm as evaluated by the blind tip reconstruction approach using a titanium roughness sample (RS-15 M, Bruker, Germany). The estimated normal load that was applied on a sample by the tip is:

$$F_N = V_{\text{setpoint}} \cdot \text{Sensitivity} \cdot K_N$$  \hspace{1cm} (1)

where $V_{\text{setpoint}}$ is the voltage of the laser on the photodetector that was set before the
scratching process. The sensitivity of the light lever system can be obtained from the slope of the force–distance curve when the tip presses onto a hard sapphire sample (SAPPHIRE-15 M, Veeco, USA). The normal loads that were used in this study ranged from 50 μN to 300 μN with a spacing of 50 μN. The samples that were used in this study were single-crystal silicon (100) wafers (Suzhou Research Semiconductor Co., Ltd., China), which were polished on a single side. The measured surface root-mean-square roughness of the sample was ~0.1 nm by scanning a 1 μm × 1 μm area using the AFM. To compare the effects of tip-based static and UVS on the subsurface damage, three methods for nanochannel fabrication that were investigated in detail are static scratch (SS) (Figure 1(f)), UVS (Figure 1(g)), and double-pass scratch (Figure 1(h)). These three nanochannel fabrication approaches are described below.

(1) SS approach

As shown in Figure 1(f), the AFM system was operated in Nanoman mode and the tip was set to machine the sample only once. In the SS approach, no driving signal was generated from the signal generator during machining, thus, the sample was static during the scratching. The scratch direction was selected as the side-forward direction along the [ 1 ̅ 1 0 ] crystal orientation.

(2) UVS approach

Ultrasonic vibration was applied in the vertical direction with a piezoelectric actuator under the drive of a sinusoidal signal (Figure 1(g)). The dynamic behavior of the piezoelectric actuator was investigated based on a microscope and microparticles. Some microparticles were placed on the sample surface to observe their motion by
using the microscope when the piezoelectric actuator conducted ultrasonic vibration.

By monitoring the microparticle motion and distribution, an appropriate excitation frequency can be found that ensures that the sample is vibrated with a maximum vibrational amplitude, which was 0.2 MHz in this study. The vibration amplitude of the piezoelectric actuator was 1.5 nm as tested by the AFM system. The excitation frequency of the driving signal was larger than the resonance frequency of the tip cantilever, and thus, the cantilever dynamically ‘freezes’ because of its inertia and the tip does not vibrate with the sample during UVS [24]. The tip pressed into the sample with a preset normal load and scratched along a linear trajectory while the piezoelectric actuator produced an ultrasonic vibration.

(3) Double-pass scratch approach

Figure 1(h) provides a schematic diagram of the double-pass scratch process. The double-pass scratch approach was carried out using the Nanoman mode of the AFM system. Two different machining methods—the SS and UVS—were used in the first scratch process. When the first scratching finished, the tip returns to the initial position under PZT control in tapping mode, which guarantees an interaction force between the tip and sample to protect the nanochannel morphology. The AFM system changed to contact mode again to repeat the SS process during the second scratch.

The morphologies of the obtained nanochannels were measured by SEM (Quanta 200FEG, FEI, USA) and AFM (Dimension Icon, Bruker, Karlsruhe, Germany). The SEM measurement was conducted immediately after scratching without further treatment. To remove the generated chips, the sample was scanned with a silicon AFM
tip in contact mode with a relatively small normal load and cleaned ultrasonically in alcohol for 10 min. A new silicon tip (TESPA, Bruker, Germany) was used to measure the topographies of the nanochannels in tapping mode. A commercial Renishaw Raman system (InVia-Reflex, Renishaw, England) was used to characterize the material structural changes. The laser wavelength was 633 nm and the laser beam power was 1.7 mW. The objective lens and typical accumulation parameters were 100× and 10 s, respectively. The subsurface characteristics of the machined structures were observed by transmission electron microscopy (TEM) (FEI Tecnai F20, USA). Cross-sectional TEM samples were prepared by using a focused-ion beam (FIB, FEI Helios NanolabG3 UC, USA), and the samples were thinned to ~90 nm to enable electron transmission. A protected layer (Pt) on the machined nanostructures was coated before TEM sample preparation to avoid sample surface and subsurface damage that was induced by FIB.

Figure 1. (a) Schematic of AFM-based UVS setup, (b) SEM micrograph and (c)
schematic of diamond AFM tip. (d,e) Schematic diagrams of side and front views of diamond tip. Schematic representation of (f) static scratch, (g) ultrasonic vibration-assisted scratch, and (h) double-pass scratch.

2.2 MD simulation model

Figure 2 shows the MD simulation model of the UVS approach. The inclination angle (~12°) of the tip was subject to the mounting angle of the tip cantilever. The single-crystal silicon substrate contained 129,271 Si atoms and a single diamond AFM tip was modeled as a rigid body. The feature dimensions of the substrate were 38 nm × 22 nm × 14 nm. The Si atoms were arranged in a diamond cubic lattice structure with a lattice constant of 5.428 Å. As shown in Figure 2, the workpiece was categorized into three parts: boundary, thermostat, and Newtonian atoms. Boundary atoms were used to reduce the boundary effect and maintain the proper lattice symmetry. Thermostat atoms were kept at a constant 293 K. The Stillinger–Weber potential function was used to calculate the interatomic forces between the Si–Si atoms, which was an appropriate potential for the simulation of covalent bonding materials [26]. The interaction between the tip and sample atoms (C–Si) was expressed by the Morse potential [27]. The initial velocities of the Si atoms were assigned in accordance with the Maxwell–Boltzmann distribution and the model was equilibrated to 293 K under the canonical ensemble (NVT) in 50 ps. The vibrational amplitude of the sample and tip feed rate were 5 Å and 2 m/s. The vibrational frequency was 20 GHz in this model to depict the sample vibration. Other simulation details of the model are listed in Table 1. A large-scale atomic/molecular massively parallel simulator was used to conduct the MD simulation [28], and the simulation results were visualized using OVITO software [29].
Table 1 MD simulation parameters

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Value</th>
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<tr>
<td>Workpiece size</td>
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</tr>
<tr>
<td>Atoms in workpiece</td>
<td>129271</td>
</tr>
<tr>
<td>Atoms in tool</td>
<td>4427</td>
</tr>
<tr>
<td>Feed rate</td>
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</tr>
<tr>
<td>Machining distance</td>
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<tr>
<td>Amplitude</td>
<td>5 Å</td>
</tr>
<tr>
<td>Equilibration temperature</td>
<td>293 K</td>
</tr>
<tr>
<td>Time step</td>
<td>1 ps</td>
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3. Results and discussion

3.1 Investigation of material removal state

As a hard brittle material, single-crystal silicon will be removed in the brittle regime and cracks, material peeling, or debris will be induced when the machined depth reaches a critical value for the brittle-to-ductile transition depth [19]. Thus, the material removal state is key for studying nanomachining on single-crystal silicon. Figure 3 shows the normal load-horizontal force-distance curves that were acquired in the SS and UVS tests at 3 μm/s, where the range of the normal load was set from 0 μN to 300 μN. Curves (a) and (b) in Figure 3 show the relationship between the horizontal force and the scratching distance when scratching using the SS and UVS approach,
respectively. The horizontal force for SS was larger than that for UVS. By comparing the horizontal forces in enlarged images I and II in Figure 3, it can be seen that the fluctuation in SS exceeds that in UVS. Curve (c) represents the applied normal load during scratching. Inserted images III and VI in Figure 3 show the SEM images that correspond to nanochannels that were machined by SS and UVS, respectively. An inspection of the horizontal force curves (a) and (b) that were recorded during loading and the SEM images indicates that the SS and UVS processes can be separated into three regimes in sequence: rubbing, plowing, and cutting. The material chips that were generated in the cutting regime for SS and UVS were visible in enlarged SEM images V and VII. For the SS process, materials were removed by rubbing, plowing, and cutting when the scratch distance was 0–9 μm, 9–28 μm, and 28–70 μm, respectively. However, for the UVS process, the scratch distance of the three regimes was 0–4 μm, 4–15 μm, and 15–70 μm, respectively. Therefore, the UVS contributes to a shorter scratch distance of the rubbing and plowing compared with the SS. Figure 4(a) and (b) shows the AFM image and cross section that corresponds to the end of the machined nanochannel (III) in Figure 3. The inserted SEM image in Figure 4(a) reveals the nanochannel morphology that is marked in a dotted yellow rectangle. Figure 4(b) shows that the machined depth at the end of the obtained nanochannel was ~60 nm. No cracks and other brittle fracture were visible in Figure 4(a) [30], which means that the sample materials were expelled in the ductile regime when the applied normal load ranged from 0 to 300 μN.
Figure 3. Nanoscratching tests with increasing normal load. (a,b) Horizontal force recorded during SS and UVS, respectively. (c) Normal load along a scratching length of 70 μm. Inserts display SEM images that correspond to the fabricated nanochannel.

Figure 4. (a) AFM image and (b) cross section of nanochannel end in Figure 3 (III).

3.2 Nanochannel morphology
The effect of scratching approaches, such as SS and UVS, on the machined nanochannel morphology was studied by SEM and AFM. Figure 5(a) and (b) provides an SEM image of the nanochannel that was fabricated by SS and UVS with normal load of 200 μN, respectively. The enlarged image in Figure 5(a) shows that a long curved segmental chip was generated at the end of the nanochannel. In contrast, the chip that formed at the end of the nanochannel that was fabricated by using UVS was a granular chip (Figure 5(b)). The inserted schematic diagrams in Figure 5(a) and (b) depict the material removal state that was fabricated by using the SS and UVS approaches, respectively. The observed SEM results show that the chip that was generated by using SS was longer than that formed by UVS. For the UVS process, a simplified elastic contact model was used to investigate the tip-sample interaction in the previous studies [25,31]. The relationship between the interaction force and tip-sample distance \(d\) can be calculated as follows:

\[
F(d) = \frac{E^* A^3}{R} - \left(\frac{3\pi}{2} \sigma E^* A^3\right)^{1/2} - F_{\text{att}}
\]

(2)

where \(R\) is the radius of the tip, and \(d\) is the distance between the tip and sample. \(\sigma\) is the adhesion energy defining as the work per unit area of contact required to separate two solid surfaces, which is assumed as 360 mJ/m\(^2\). The long-range attraction force \(F_{\text{att}}\) can be neglected in this study [32]. \(A\) is the radius of the tip-sample contact area and it can be estimated by \(A^2 \approx R d\). Thus, the Eq. (2) can be further expressed as follows:

\[
F(d) = E^* (Rd^3)^{1/2} - \left(\frac{3\pi}{2} \sigma E^* (Rd)^{3/4}\right)^{1/2} - F_{\text{att}}
\]

(3)

The effective elasticity \(E^*\) can be expressed as follows:
where $E_t$ and $\nu_t$, $E_s$ and $\nu_s$ are Young’s modulus and Poisson’s ratio of the tip and sample, respectively, which is 1120 GPa, 0.12 [33], 127 GPa and 0.25 [34]. Applying Eqs (3) and (4), the force–distance relationship can be obtained as shown in Figure 5(c). $F_N$ in Figure 5(c) is the setting normal load which is 200 $\mu$N in this study, the tip–sample distance ($d_1$) corresponding to the $F_N$ is calculated to be 44.6 nm. The amplitude of the ultrasonic vibration ($a$) is 1.5 nm, as shown in Figure 5(c), which can introduce $\pm 1.5$ nm change into tip–sample distance. Thus, the maximum tip–sample distance can be obtained by $d_2=d_1+1.5$, which is 46.1 nm. According to Eq. (3), the interaction force between tip and sample at the maximum tip–sample distance ($F_p$) is calculated to be 219 $\mu$N. The energy applied by the tip during nanoscratching can be obtained from the interaction force between the tip and sample multiplying the cutting depth, which can be estimated as follows [35]:

$$E = F_p \cdot D$$  \hspace{1cm} (5)

where $F_p$ and $D$ is the interaction force and machined depth, respectively. In this study, the machined depth for SS and UVS is 50 nm and 60 nm respectively when the setting normal load is 200 $\mu$N. Applying Eqs (3)-(5), the calculated energy that applied by the tip for UVS ($E_{UVS}$) and SS ($E_{SS}$) is $1.314 \times 10^{-8}$ mJ and $1 \times 10^{-8}$ mJ, respectively. Thus, the energy applied by UVS ($E_{UVS}$) is approximately 1.3 times of that for SS ($E_{SS}$). The higher input energy that was caused by ultrasonic vibration in UVS may contribute to the fracturing of the formed chips compared with the SS process. The AFM images of the nanochannels that were fabricated by using SS and UVS with a normal load of 200
μN are shown in Figure 5(d) and (f). There was almost no pile-up for the obtained nanochannels. Figure 5(e) and (g) shows the cross-sections that correspond to the nanochannels as shown in Figure 5(d) and (f), respectively. The experimental results show that the nanochannel depth that was obtained by using UVS was deeper compared with that fabricated by the SS approach.
Figure 5. SEM images of nanochannels fabricated using (a) SS and (b) UVS at 0.2 MHz. (c) The interaction force between the tip and sample. AFM images and corresponding cross sections of nanochannels fabricated using (d,e) SS, (f,g) UVS of 0.2 MHz.

To understand the effect of the machining approaches on the nanochannel
dimensions, we conducted the SS and UVS processes with a normal load from 50 to 300 μN and a spacing of 50 μN. Figure 6(a) shows that the machined depth increased with an increase in applied normal load for scratching by using SS and UVS. Figure 6(a) shows that the machined depth of the UVS was deeper than that of the SS at the same applied normal load. The workpiece underwent an ultrasonic vibration with a given amplitude for UVS, which led to a relative motion between the tip and workpiece in the vertical direction during scratching. Thus, the machined depth was deeper in this scenario compared with scratching by SS. Similarly, the nanochannel depth that was scratched by using UVS was deeper than that when using SS when machining with the same tip feed rate (Figure 6(b)). Figure 6(b) shows that the machined depth decreased with an increase in feed rate for the SS and UVS approach. A higher feed rate resulted in a relatively large sample material strain rate during scratching, which increased the contact pressure between the tip and sample [36]. Thus, the machined depth was shallow at a larger feed rate to balance the constant normal load.

Figure 6. Relationship between machined depth of nanochannels and (a) applied normal load, (b) tip feed rate when machining using SS and UVS approaches.

3.3 Nanochannel phase transformation
Figure 7(a) shows the Raman spectra of the nanostructures that were machined by using SS and UVS. Pristine nanostructures were used compared with the machined nanostructures. Three characteristic peaks in the SS and UVS spectrums were visible, and included two broadband peaks at 150 cm\(^{-1}\) and 470 cm\(^{-1}\) and a strong and sharp peak at 521 cm\(^{-1}\). These characteristic peaks can be identified as the \(\alpha\)-Si phase and Si-I phase [37]. A series of phase transformations of single-crystal silicon occur during machining [38]. The Si-I phase of the single-crystal silicon transforms into metallic Si-II phase because of the high pressure. The metallic Si-II is unstable and will transform into other phases. The transformation is determined by the rate of pressure release. The Si-II phase will transform into Si-III and Si-XII phases at a relatively low rate of pressure release, and into an \(\alpha\)-Si phase when the release rate is high [39]. In this study, the Raman spectra indicate that a layer of amorphous silicon was induced in the SS and UVS process owing to the higher rate of pressure release.

Figure 7(a) shows that the Raman intensity of the UVS is stronger than that of the SS. The Raman intensity is related to the amorphous layer thickness of single-crystal silicon [37]. A stronger Raman intensity yields a thicker amorphous layer [40]. Thus, the Raman spectra reveal that the amorphous layer that is induced by UVS is thicker than that by SS. To understand the phase transformation of SS and UVS, we used an MD simulation approach to simulate the scratch process. An analysis of the range of phase transformations was undertaken by the coordination number \((n)\) in the workpiece. Different phases of single-crystal silicon, including the diamond crystal phase, bct-silicon, and \(\beta\)-silicon, were identified based on the coordination numbers.
Figure 7(b) and (c) shows the simulated phase transformation of SS and UVS, respectively. The contact area (I in Figure 7(b)) between the tip and sample was composed mainly of metastable atoms \( n = 7 \) mixed with bct-silicon \( n = 5 \) and \( \beta \)-silicon \( n = 6 \), which was generated from the high pressure. The contact region for UVS (area III) was thicker than that for SS (area I). The bottom region of the nanochannel (II in Figure 7(b)) was composed of amorphous silicon atoms. The atoms were mainly bct-silicon \( n = 5 \) and \( \beta \)-silicon \( n = 6 \), which transformed from metastable atoms when the tip moved away and from the effect of unloading. A comparison of the bottom region of the nanochannel scratched by using SS (II in Figure 7(b)) and UVS (IV in Figure 7(c)) showed that region IV was thicker. The MD simulations revealed that the amorphous layer that was induced by UVS was thicker, which agrees well with the Raman spectra.

3.4 Subsurface damage of nanochannels

We used TEM to investigate the subsurface damage mechanism of single-crystal
silicon during ductile-machining using the SS and UVS approach. As shown in Figure 8(a) and (b), cross sections of the nanochannels that were scratched by SS and UVS, which were termed channels I and II, respectively, were detected by TEM. A layer of amorphous silicon was induced beneath the machined nanochannel, evidenced by the diffraction pattern in Figure 8(a) [41,42]. Figure 8(a) and (b) shows that the amorphous layer for channel II is thicker than that for channel I. The results indicate agreement between the Raman spectra and TEM observations. The machined depths for channel I \(d_1\) in Figure 8(a)) and II \(d_2\) in Figure 8(b)) are identical at \(~80\ nm. Furthermore, the depths of subsurface damage for channel I \(D_1\) and II \(D_2\) are \(~70\ nm and 140\ nm, respectively. The TEM observation indicates that the UVS will induce a larger subsurface damage compared with the SS approach.

To understand what happened in the subsurface, we performed high-resolution TEM (HRTEM) in the regions beneath the induced amorphous layer, as pointed out in Figure 8(a) and (b). The results that correspond to the selected areas in Figure 8(a) and (b) are shown in Figure 8(c)–(i). We selected a region (c in Figure 8(a)) far from the affected area as a reference to prove that subsurface damage was induced by the scratch. As shown in Figure 8(c), no crystal defects existed in the pristine material. The diffraction pattern that corresponds to the region, which is also provided in Figure 8(c), illustrates strong lattice diffraction. The result indicates a single-crystal structure [21]. Hence, on the basis of the HRTEM and diffraction pattern of the pristine material, the sample was single crystal without defects before machining, and any subsurface damage after machining was caused by scratching.
Figure 8(d) shows the HRTEM image of the area (d in Figure 8(a)). Dislocations and stacking faults were induced for channel I during SS. The high pressure and shear stress that were generated by SS contribute to these crystal defects [43]. Figure 8(f)–(i) presents the HRTEM images that correspond to the marked areas in Figure 8(b). Dislocations and stacking faults are also visible in Figure 8(f), furthermore, the enlarged image (Figure 8(e)) of the area in Figure 8(f) is indicative of amorphous silicon [21]. The rapid release of pressure and an increase in defect concentration, such as dislocations and stacking faults, facilitate amorphous phase formation [39, 44]. HRTEM images of the subsurface of channel II in Figure 8(g) and (h) reveal that in addition to dislocations, stacking faults and amorphous silicon are induced, distortion of atomic plane (Figure 8(g)) and slip band (Figure 8(h)) do exist in the plastic-deformation zone [45,46]. Figure 8(i) shows atomic-scale defects, including dislocations, stacking faults, and amorphous silicon. The HRTEM results demonstrate that the plastic deformation mechanism under SS is different with UVS. For the SS approach, only pressure-induced defects, such as dislocation and stacking faults were observed. However, for UVS, in addition to these defects, a small amount of amorphous phase, slip band, and distortion of atomic plane was identified in the damage area. Furthermore, the depth of subsurface damage that was induced by UVS was deeper than that for the SS.
Figure 8. TEM images of nanochannels fabricated using (a) SS and (b) UVS. (c, d) Enlarged image of area marked in (a). (e) Enlarged image of area marked in (f). (f)–(i) Enlarged images of area in (b).

We used a MD simulation approach to simulate the SS and UVS processes and understand why the subsurface damage that was induced by UVS was deeper than that by SS. We identified dislocations and their Burgers vectors with the aid of a dislocation extraction algorithm. Figure 9(a) and (b) shows the distribution of dislocations from MD simulation of SS and UVS, respectively. The identified dislocations with Burgers vectors $\frac{1}{2}[110]$, $\frac{1}{6}[112]$ and unidentified dislocations were colored blue, green, and red, respectively. Many dislocations are visible in Figure 9(a) and (b) beneath the machined channel. The MD simulation revealed that the full length of the dislocation for SS and UVS was 519 nm and 675 nm, respectively. The area that was affected by dislocation for UVS was deeper than for SS, which agrees with the TEM results. We recorded the variation in potential energy for the MD simulation system, as shown in Figure 9(c). The potential energy of the simulation system increased with tip advancement, which indicates that the sample experiences different degrees of deformation during scratching [47]. Figure 9(c) also shows that the potential energy for UVS is larger than that for SS when the tip advancement exceeded 7 nm because of the higher input energy that was generated from the ultrasonic vibration in UVS compared with the static scratching. A previous study reported that the cutting energy can be separated into heat-generation, surface-generation, and material-disorder energy. The material-disorder energy contributes to subsurface damage, such as dislocation and stacking faults [48]. The temperature of the SS and UVS simulation system was
constant at 293 K. Thus, we eliminated the effect of heat-generation energy in this study.

The separation energy for new surface generation can be calculated from \( E = e\gamma \left( \frac{1}{h} \right) \),

where, \( e \), \( \gamma \), and \( h \) are the surface energy of the sample material, relief angle of the tip, and the undeformed chip thickness, respectively [48]. The undeformed chip thickness for SS was the same as that for UVS for an identical machined depth. Because the \( e \), \( \gamma \), and \( h \), were the same, there was no difference in surface generation energy for SS and UVS. Thus, more material disorder energy was exerted on the workpiece for UVS than SS and resulted in a relatively large subsurface damage.

According to the TEM and MD simulation, we proposed schematic diagrams of subsurface damage for nanochannels that were fabricated by using SS and UVS, as shown in Figure 9(d) and (e). SS and UVS yielded a layer of amorphous silicon during machining. Dislocation and stacking faults were present in the subsurface of the channels that were machined by SS and UVS because of the high pressure and shear stress. More atomic defects, such as atomic-plane distortion, local amorphization, and slip bands were induced in the UVS (Figure 9(e)). Owing to a higher material disorder energy, the subsurface damage that was caused by UVS (Figure 9(e)) was deeper than SS (Figure 9(d)). The subsurface damage from UVS at the nanoscale was inconsistent with that caused by the UVM at the macro- or microscale. UVM at the macro- and microscale facilitated shallow subsurface damage compared with conventional static machining [10-13]. The sample material during machining experiences plastic deformation before it fractures. The plastic deformation is controlled by dislocation motion. For macro- and microscale-UVM, the embrittlement of sample material that
was caused by dislocation immobilization and dislocation avalanche was regarded as the reason for a relatively shallow subsurface damage [14]. However, for nanoscale-UVS, the induced dislocations were less than those generated by microscale-UVM. The mobile dislocations extended inside the sample material more easily compared with the microscale-UVM, rather than being trapped and converted to immobile dislocations that distribute close to the machined surface. Thus, the material was more plastic during nanoscale-UVS compared with macroscale-UVM, which led to a relatively deep subsurface damage. Our experimental results indicate that the UVM was unsuitable for machining when the machined depth reached the nanoscale when aiming to suppress the subsurface damage.

Figure 9. Distribution of dislocations obtained by MD simulation of single-crystal silicon
machining with (a) SS and (b) UVS. (c) Potential-energy evolution versus tip-movement distance obtained by MD simulation. Schematic diagrams of subsurface damages for nanochannels fabricated using (d) SS and (e) UVS, respectively.

3.5 Effect of subsurface damage on machining mechanism

To investigate the influence of subsurface damage on the machining mechanism, we repeated the scratch process on the already machined nanochannels, which was termed double-pass scratching in this study. The experimental details of the double-pass scratching were introduced in Section 2.1. Two kinds of double-pass scratch approaches were conducted: (i) the first and second scratches were both achieved by static scratch and was defined static-static scratching (SS–SS) and (ii) the first and second scratches were achieved by UVS and static scratching, respectively, and was termed UVS-static scratching (UVS–SS). Figure 10(a) and (b) shows the SEM images of the nanochannels that were machined by SS–SS and UVS–SS. Curved chips (indicated by red arrows) formed during the second scratch process in Figure 10(a) and (b). The long curved segmental chips (indicated by green arrows) in Figure 10(a) and the granular chips in Figure 10(b) were generated during the first scratch, as discussed in Section 3.2. A comparison of the curved chips with the chips that were generated in the first scratch showed that the sample material plasticity improved after the first scratch [49]. The expelled materials in the second scratch process were mainly amorphous silicon and single-crystal silicon with many atomic defects. The amorphous silicon and atomic defects, such as dislocation and stacking faults, facilitated material removal in the plastic regime during the second scratch [50-52].

Figure 10(c) shows the relationship between the machined depth of the
nanochannel that was fabricated by using SS–SS and UVS–SS and the applied normal load. Therefore, the machined depth increased linearly with an increase in applied normal load. The machined depth of the SS–SS was almost identical to that of the UVS–SS. We discussed the machined depth of the SS and UVS in Section 3.2 and reported that the machined depth of the UVS was deeper than that of the SS. We calculated the machined depth during the second scratch process, as shown in Figure 10(d). The machined depth during the second scratch for SS–SS was larger than that for UVS–SS. A previous study has shown that the sample material is significantly harder than the pristine material after plastic deformation owing to the induced dislocations [48]. In the first scratch process, the TEM results revealed that more atomic defects resulted from UVS than SS, which led to a harder sample material for UVS–SS compared with SS–SS in the second scratch. Therefore, the machined depth in the second scratch for SS–SS was large compared with the UVS–SS. On the basis of the analysis, we provided schematic diagrams of the double-pass scratching for SS–SS and UVS–SS in Figure 10(e) and (f), respectively. In the first scratch, the machined depth for the SS–SS ($m_{ss}$ in Figure 10(e)) was smaller than that for UVS–SS ($m_{uv}$ in Figure 10(f)). However, in the second scratch, owing to the effect of subsurface damage that was induced by the first scratch, the machined depth for the SS–SS ($M_{ss}$) was larger compared with the UVS–SS ($M_{sb}$). The total machined depth of the nanochannel that was fabricated by the two double-pass scratch approaches was approximately identical.
Figure 10. SEM images of nanochannels fabricated using double scratching approach: (a) SS–SS, (b) UVS–SS. (c) Relationship between machined depth and normal load when machining using double scratching. (d) Machined depth in second machining process for different normal loads. Schematic diagrams of subsurface damage for (e) SS–SS and (f) UVS–SS, respectively.

4. Conclusions

We investigated the subsurface damage mechanism of hard–brittle material (i.e.,
single-crystal silicon) during ductile-machining using UVS. The investigation involved a study of the material removal state, morphology, and dimensions of the machined nanochannel, and the effect of subsurface damage on the scratch outcomes. Our major findings were given as follows:

- Single-crystal silicon was removed by rubbing, plowing, and cutting in sequence with an increase in normal load. Compared with conventional static nanoscratching, the UVS led to a deeper machined depth and a shorter chip.

- The TEM, Raman spectroscopy, and MD simulation demonstrated that the subsurface damage caused by UVS was deeper than that caused by static nanoscratching owing to a larger material disorder energy. Compared with macro- and microscale UVM, the material was more plastic during nanoscale-UVS, which led to an opposite phenomenon of subsurface damage.

- The UVS resulted in more atomic defects, such as local amorphization, slip band, and distortion of the atomic plane in addition to dislocation and stacking faults compared with static nanoscratching.

- By using the double-pass scratch approach, we verified that the silicon was significantly harder than the pristine material after plastic deformation. The machined depth in the second scratch for SS–SS was relatively large compared with the UVS–SS owing to fewer induced dislocations.

This work showed the subsurface damage mechanism of single-crystal silicon during ductile-machining using UVS. Different from the microscale UVM, we found that the nanoscale UVM induced deeper subsurface damage compared with conventional static machining. Therefore, we believe this could provide an instruction that UVM may be not a good choice to suppress subsurface damage of hard–brittle materials (such as...
silicon) when the machined depth reaches to the nanoscale.

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References


[34] X. Li, Y. Gao, P. Ge, L. Zhang, W. Bi, J. Meng, Nucleation location and propagation direction of radial and median cracks for brittle material in scratching, Ceramics International 45 (2019) 7524-
1 7536.
2 [35] Z. Lin, Y. Hsu, A calculating method for the fewest cutting passes on sapphire substrate at a
certain depth using specific down force energy with an AFM probe, Journal of Materials Processing
3 [36] C. Li, F. Zhang, Y. Wu, X. Zhang, Influence of strain rate effect on material removal and
deformation mechanism based on ductile nanoscratch tests of Lu2O3 single crystal, Ceramics
International 44 (2020) 21486-21498.
4 [37] J. Yan, T. Asami, H. Harada, T. Kuriyagawa, Fundamental investigation of subsurface damage in
5 [38] Y. Wu, H. Huang, J. Zou, L. Zhang, J. Deld, Nanoscratch-induced phase transformation of
2858-2869.
7 [40] J. Yan, T. Asami, T. Kuriyagawa, Nondestructive measurement of machining-induced amorphous
layers in single-crystal silicon by laser micro-Raman spectroscopy, Precision Engineering 32 (2008)
186-195.
amorphous Si solar panels, International Journal of Machine Tools & Manufacture 51 (2011) 797-
805.
9 [42] X. Rao, F. Zhang, Y. Lu, X. Luo, F. Chen, Surface and subsurface damage of reaction-bonded
silicon carbide induced by electrical discharge diamond grinding, International Journal of Machine
Tools & Manufacture 154 (2020) 103564.
11 [44] Z. Shi, J. Zhang, Q. Zhao, B. Guo, H. Wang, Transmission electron microscopy (TEM) study of
anisotropic surface damages in micro-cutting polycrystalline aluminate magnesium spinel (PAMS)
12 [45] C. Li, X. Li, Y. Wu, F. Zhang, H. Huang, Deformation mechanism and force modelling of the


