

## Effect of temperature and stress on plastic deformation in monocrystalline silicon induced by scratching

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Dry air, coolant, and liquid nitrogen were applied, respectively, to study the effect of temperature and stress on plastic deformation in scratching monocrystalline silicon. Phases generated in surface deformation were characterized by means of the transmission electron microscopy. It was shown that the size of the amorphous transformation zone and the depth of slip penetration in sample subsurface were mainly dependent on the stress field applied. The influence of the temperature variation to  $-196\text{ }^{\circ}\text{C}$  was surprisingly small and the low temperature did not suppress the phase transformation and dislocation activity. © 2005 American Institute of Physics.

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Silicon is a fundamental material for electronic and photovoltaic technologies and as such an in-depth understanding of its deformation mechanisms under a wide variety of external conditions are of significant importance.<sup>1-3</sup> Monocrystalline silicon is known as a brittle material at temperatures below  $0.5T_m$  ( $T_m$  is the melting temperature of silicon);<sup>4</sup> however limited plasticity featuring phase transformation to amorphous or crystalline metastable R8/BC8 (a body-centered cubic/rhombohedral<sup>5</sup>) phases and dislocation activities are widely admitted under complex loading in different surface modification processes such as indentation,<sup>6-17</sup> scratching,<sup>18-22</sup> turning,<sup>23-27</sup> grinding, and polishing.<sup>13,16,25,28-31</sup> In all mentioned processes material deformation is associated with some degree of local temperature rise.<sup>16,32</sup> Therefore it is hard to separate stress and temperature contributions to the micro plastic deformation of silicon.

In an attempt to eliminate the possible temperature rise, Khayyat *et al.* performed indentation experiments in liquid nitrogen.<sup>32</sup> No phase transformation was detected inside the residual indentation marks by means of Raman spectroscopy, which led to an impression that structural transformations at 77 K were unlikely to happen.<sup>32</sup> On the other hand, a number of studies confirmed negligible temperature rise in samples undergoing phase transformation.<sup>18,23</sup> In addition, no correlation between the feed velocity and the amount of phase transformation was admitted in grinding experiments, which indirectly indicated that the temperature rise played a minor role.<sup>28</sup> It is therefore still unclear if the phase transformations and dislocations can be activated in silicon without a substantial temperature rise.

In this study we intend to clarify the problem by conducting scratching experiments in dry air, coolant, and liquid nitrogen, respectively. The phase transformations will be identified by means of a cross-section view technique<sup>12</sup> and transmission electron microscopy.

The scratching tests on the (100) surfaces of monocrystalline silicon along  $\langle 100 \rangle$  direction with a fixed normal load  $P=100\text{ mN}$  were performed on a modified reciprocating scratching machine with a conical diamond indenter having a cone angle of  $120^{\circ}$  and a nominal tip radius of  $8.2\text{ }\mu\text{m}$ . The

scratching velocity was  $2.67\text{ mm/s}$  and the scratched length was  $20\text{ mm}$ . Before a scratch test, the wafer surface was polished in ultraprecision mode to ensure that no subsurface damage took place.<sup>12</sup> The dry scratching tests were conducted in open air. In the tests with coolant, water based coolant was spread over the sample's surface before scratching. In order to perform a test at a temperature of  $-196\text{ }^{\circ}\text{C}$ , a sample was placed in a polystyrene container filled up with liquid nitrogen. The sample and indenter were completely immersed in the liquid nitrogen for 20 min before scratching. After the experiment, liquid nitrogen was allowed to evaporate and the sample rose to the room temperature gradually.

The topography of the scratches was studied by means of an atomic force microscope. TEM studies were carried out in a Philips CM12 transmission electron microscope operating at 120 kV. The  $\langle 110 \rangle$  cross-section TEM specimens were prepared using a tripod.<sup>15</sup> In the preparation, the sample surface was kept perpendicular to the scratching direction.<sup>33</sup> Finally, the ion-beam thinning was carried out to provide a sufficiently thin area for the TEM investigations.

Figure 1 presents topography of scratches obtained in three cooling media of dry air, water based coolant, and liquid nitrogen. Microcracks were evident on the samples' surface scratched in dry air. The scratch generated in air was  $6\text{ }\mu\text{m}$  in width and  $320\text{ nm}$  in depth [Fig. 1(a)]. Bumps could clearly be seen near the scratch edges, indicating that a phase transformation to a ductile phase during stressing occurred.

In contrast, no surface microcracks were observed when the tests were done in coolant. Scratches appeared with the same width ( $6\text{ }\mu\text{m}$ ) but with a much shallower depth ( $180\text{ nm}$ ) [Fig. 1(b)]. The scratch bottom was rather rough in this case. Bumps were also clearly distinguished near the scratch edges, confirming an extrusion of a ductile phase.

The scratch geometry was altered largely when the tests were in liquid nitrogen. The scratch width reduced to  $5\text{ }\mu\text{m}$  and the depth to  $120\text{ nm}$  [Fig. 1(c)]. The bottom of the scratch was rough with some brittle fragments and extensive microcracks were developed on the sample surface. Surprisingly, the bumps still exist near the scratch edges, which seems to suggest a phase transformation at the temperature of  $-196\text{ }^{\circ}\text{C}$ .

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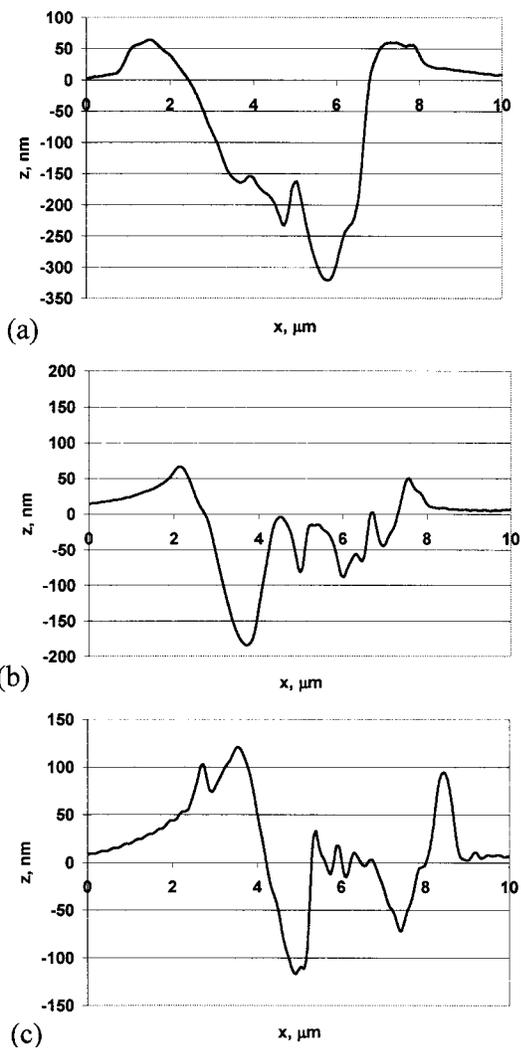


FIG. 1. Scratch profiles in different cooling media: (a) dry air, (b) water based coolant, (c) liquid nitrogen.

Figure 2 shows the diffraction contrast images, demonstrating the morphology and microstructure of the transformation zone after scratching experiments in the three media, where the diffraction patterns were inserted to show the crystallinity inside the transformation zone. It is clear that transformation zones developed in all three media, but the amount and feature of the transformed materials were dissimilar. The scratching in dry air [Fig. 2(a)] generated mainly amorphous transformation zone [see the inset in Fig. 2(a)] with its width and depth of 6  $\mu\text{m}$  and 450 nm, respectively. Large microcracks were developed beneath the zone, but dislocation activities were limited, probably due to the extensive microcracking.

The scratching with coolant generated a shallower transformation zone (depth = 360 nm and width = 6  $\mu\text{m}$ ) [Fig. 2(b)], which is mainly amorphous [see the inset in Fig. 2(b)]. However, small crystalline areas near the bottom of the zone were identified as diamond silicon. The slip line penetration was evident to the depth of 1.6  $\mu\text{m}$ . Also, the median crack developed at sites of slip line interactions.

The subsurface structure after scratching in liquid nitrogen [Fig. 2(c)] indeed confirmed phase transformation as indicated by the bumps at the scratch edges [Fig. 1(c)].

The detail of the bump is presented in Fig. 3, which shows its amorphous nature (see the diffraction pattern in the

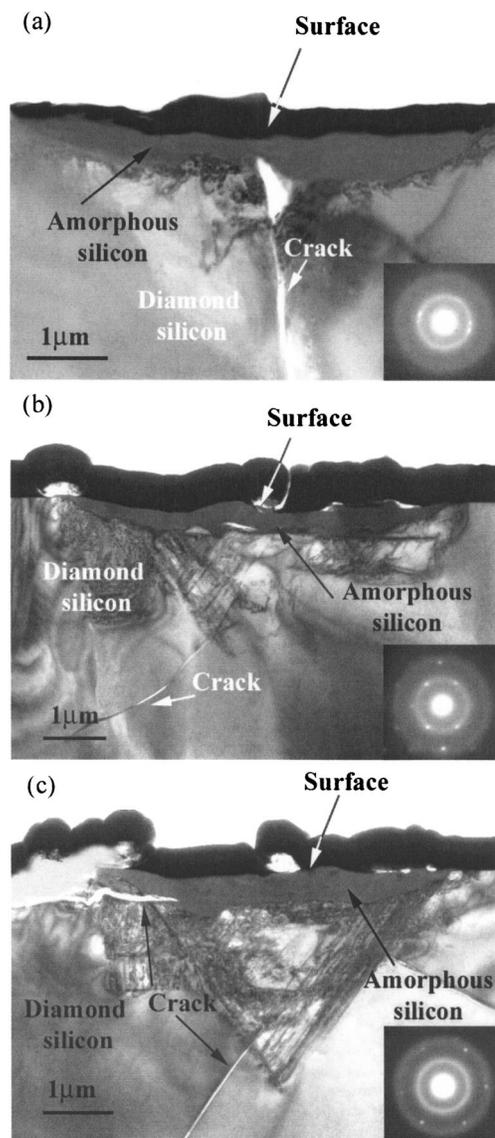


FIG. 2. The microstructure of the transformation zones after scratching in different cooling media (the diffraction patterns of the transformation zones were inserted): (a) dry air, (b) water based coolant, (c) liquid nitrogen.

inset). The height of the bump agrees with that bump presented by Fig. 1(c).

The width of the transformation zone reduced to 5  $\mu\text{m}$ , but its depth expanded to 470 nm and became almost similar to the depth of the transformation zone initiated in dry air.

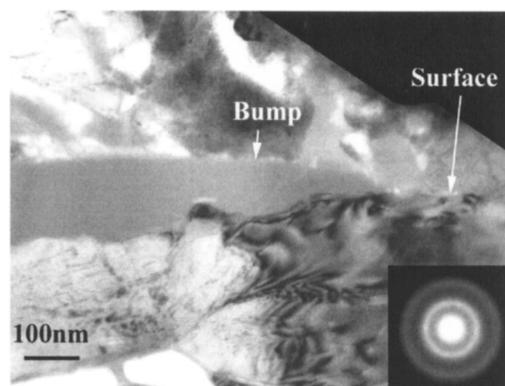


FIG. 3. The microstructure of the bump after scratching in liquid nitrogen.

Also unexpected was the extensive development of the slip systems beneath the transformation zone, which penetrated to the depth of 3.5  $\mu\text{m}$ . However, many median cracks were developed in the subsurface, emanating from the sites of slip line interactions. The arbitrary directions of the cracks indicated that diamond silicon at  $-196^\circ\text{C}$  is brittle. Besides, some amount of crystalline diamond phase was found inside the amorphous zone.

Now let us consider the details of the stress components and temperature rise in scratching experiments to try to elucidate the above noted observations. Normal forces were kept unchanged at 100 mN in all the three types of tests. The estimated tangential forces based on the coefficient of friction between silicon and diamond<sup>34</sup> were 46, 20, and 50 mN for tests in dry air, coolant, and liquid nitrogen, respectively.

To calculate the possible temperature rise during scratching, we assume that all the input energy in scratching was converted to heat and that all the heat was conducted into the silicon specimen. It is clear that an analysis based on these assumptions will overestimate the temperature rise in the specimen. For simplicity, we assume that the heat flux generated by scratching distributed uniformly in a rectangular area of  $-b/2 \leq x \leq b/2$  and  $-b \leq y \leq b$  moving along the workpiece with the scratching speed  $V$ , where  $2b$  is the groove width.

The heat conduction problem can be solved by Jaeger's model,<sup>35</sup> i.e.,

$$\bar{T}(X) = \int_{X-L}^{X+L} e^{-\eta K_0(|\eta|)} d\eta - \int_{X-L}^{X+L} e^{-\eta} d\eta \int_B^\infty \frac{e^{-(\eta^2 + \zeta^2)^{1/2}}}{(\eta^2 + \zeta^2)^{1/2}} d\zeta, \quad (1)$$

where

$$\bar{T}(X) = \frac{\pi KV}{2\chi\rho} T(X). \quad (2)$$

$K$  is the conductivity of the material,  $\rho$  is the density,  $K_0$  is the modified Bessel function of the second kind of order zero, and  $X$ ,  $L$ , and  $B$  are nondimensional parameters defined by

$$\chi = \frac{K}{\rho c}, \quad X = \frac{Vx}{2\chi}, \quad L = \frac{Vl}{2\chi}, \quad B = \frac{Vb}{2\chi}, \quad (3)$$

in which  $c$  is the specific heat. The values of the maximum temperatures occurred at the scratching surface and were determined to be 52, 37, and  $160^\circ\text{C}$  for experiments in air, coolant, and liquid nitrogen, respectively, suggesting that the temperature rises are negligible. One may argue that the heat flux generated by scratching should not be uniformly distributed as treated above. Because of the nanometer scale surface asperities on both the diamond indenter and silicon specimen surfaces, local contact can be at the size of a fraction of a nanometer. Thus the local temperature rise can be much higher. However, Zhang and Tanaka<sup>36</sup> reported that even at such a small contact size with a much higher indentation/scratch speed, the temperature rises are still low. The above discussion seems to suggest that phase transformation and dislocation initiation did take place at temperatures much lower than  $0.5T_m$ . Therefore plasticity was initiated mainly by the external stresses applied. As the normal

and tangential forces were similar in experiments with air and liquid nitrogen it is reasonable that the size of the transformation zones in these cases appeared almost identical. It is also reasonable that the smaller scratching tangential force with coolant led to a smaller depth of the amorphous zone and a shallower penetration of slip systems.

In summary, this study led to the following conclusions:

- (1) Stress field plays a major role in plastic response of silicon and the plasticity can be initiated without substantial temperature rise.
- (2) Phase transformation and dislocation activities can be initiated in monocrystalline silicon at  $-196^\circ\text{C}$ .

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