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Towards a deeper understanding of plastic deformation in mono-crystalline silicon

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Abstract

This paper investigates the plastic deformation in mono-crystalline silicon under complex loading conditions. With the aid of various characterization techniques, it was found that the mechanism of plasticity in silicon is complex and depends on loading conditions, involving dislocations, phase transformations and chemical reactions. In general, plastic deformation in silicon is the coupled result of mechanical deformation controlled by the stress field applied, chemical reaction determined by the external loading environment, and mechanical–chemical interaction governed by both the loading type and environment. Temperature rise accelerates the penetration of oxygen into silicon and reduces the critical stress of plastic yielding. When the chemical effect is avoided, the initiation of plasticity is enabled by octahedral shear stress but the further development of plastic deformation is influenced by hydrostatic stress. Plasticity of silicon in the form of phase transformations, e.g., from the diamond to amorphous or from the amorphous to bcc structures, is determined by loading history. © 2001 Elsevier Science Ltd. All rights reserved.

1. Introduction

Mono-crystalline silicon is a key material for both the micro-electro-mechanical systems and etalon components that require high surface finish and damage-free subsurface. As a result, the deformation mechanisms in the material during surface processing must be fully understood. The challenge is that in many modern surfacing processes, in addition to mechanical loading, material removal is often affected by other external factors, such as chemical reaction when polishing/grinding fluid is applied, and electric, magnetic and electronic factors when a physical field is used to assist the surfacing. Hence, to quantify the material removal process and control

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the subsurface quality, an in-depth understanding of the relationship of silicon's microstructural changes with the stresses and other fields externally applied is of great importance.

It has been widely accepted that mono-crystalline silicon has a brittle nature at ambient temperature. However, its amorphous modification exhibits a plastic flow under low loads [1] so that possible phase transformation events in machining operation are of great significance in producing damage-free components by machining. Direct and indirect experimental studies confirmed amorphous transformation and dislocation activity in ultra-precision grinding and turning [2,3]. Some traces of the amorphous phase were admitted inside the residual indentation marks but no defect was found [4–6]. An etching technique was proposed to understand the dislocation activity near scratched edges in silicon [7]. The result suggested that plasticity in scratching was caused by a pressure-induced semiconductor-to-metal phase transition [8]. Chemical reaction was emphasized in different machining operations of silicon [9–11], but the detailed mechanism has not yet been studied carefully.

This paper aims to study the mechanism of plastic deformation in mono-crystalline silicon subjected to various loading conditions.

2. Experimental details

All experiments were conducted on precisely polished (100) surfaces of mono-crystalline silicon with their rms roughness less than 2 nm. Before an experiment the subsurface structure of a specimen was examined, using the method described by Zarudi and Zhang [12], to guarantee that the specimen was damage-free.

Silicon monocrystal is a special material that behaves differently under different loading conditions. When subjected to a simple loading, a uniaxial tension for instance, it is brittle. To characterize the behavior of silicon in surface processing, it is necessary to apply complex loading conditions consistent with those in surface processing. To this end, indentation, single-point scratching, polishing and grinding were used in the present study to introduce various loading conditions. The indentation with a spherically tipped diamond indenter of radius 5 μm was made on a UMIS-2000 instrument whose details can be found elsewhere [13]. A series of tests were conducted with the indentation load of 30, 40, 50, 70, 90, and 100 mN, respectively. In each test, an 100 indentation depths were measured throughout the loading–unloading cycle. At least five indentation marks were studied at every load specified above. The indentations were carried out in a dry but open environment.

The scratching experiments at a constant speed of 20 mm/s were conducted using a Romulus III Universal Tester in $\langle 100 \rangle$ direction of silicon lattice with a spherically tipped diamond indenter of radius 300 μm scratching over a distance of 20 mm. The scratching loads normal to the silicon specimens were 2.0, 4.4, 7.8, 11.2 N, respectively. An acoustic emission system was installed to detect possible micro-cracking. The scratching experiment was also carried out in a dry and an open environment.

The grinding experiments were carried out on a modified surface grinder, the Minini Junior 90 CF CNC M286. The grinding parameters and wheel dressing conditions are listed in Tables 1 and 2, respectively. The grinder has a loop-stiffness of 80 N/ μm and a down-feed accuracy of 100 nm, which guarantees the stability and reliability of the operations.

Table 1
Grinding conditions

Grinding wheel	SD4000L75BPF
Wheel diameter (mm)	305
Wheel speed (m/s)	27
Grinding width (mm)	5
Table speed (m/min)	0.02, 0.1, 0.2, 0.5, 1
Depth of cut (nm)	100
Coolant	Sintilo 3

Table 2
Grinding wheel dressing conditions

Type of dresser	Multipoint diamond
Wheel speed (m/s)	10
Dressing cross-feed rate (mm/revolution)	0.01

Loading via polishing was conducted on a commercially available dimple-grinder, Gatan Model 656/3, whose details have been described by Zhang and Zarudi [14]. The rotation velocity of a silicon sample was 420 rpm and that of the polishing pad was 5.4 rpm. The abrasives used were α -Al₂O₃ with an average diameter of 50 nm suspended in demineralized water. The external loads applied through the polishing wheel, normal to the specimen surface of silicon of (100), were 147, 294, and 441 mN, respectively.

The subsurface structures of specimens after any of the above loading were investigated by means of TEM, CM12, and scanning transmission electron microscope (STEM), VG HB601. To fully reveal the subsurface deformation under various loading conditions, different techniques were employed. The conventional cross-section-view technique [15,16] was used for ground specimens, wedge cross-section-view method was applied for polished specimens [14], and array cross-sectioning technique was employed for the indented specimens [13].

3. Results and discussion

In the present study, plastic deformation is a generalized concept that includes the irreversible deformation caused by dislocations, planar defects, phase transformations or chemical reactions.

3.1. Deformation characteristics

The subsurface structures of silicon after indentation, scratching, grinding, and polishing are explored through Figs. 1–4, respectively. A common feature of the structures is the phase transformations in all the specimens subjected to different loading conditions, although the mechanisms vary.

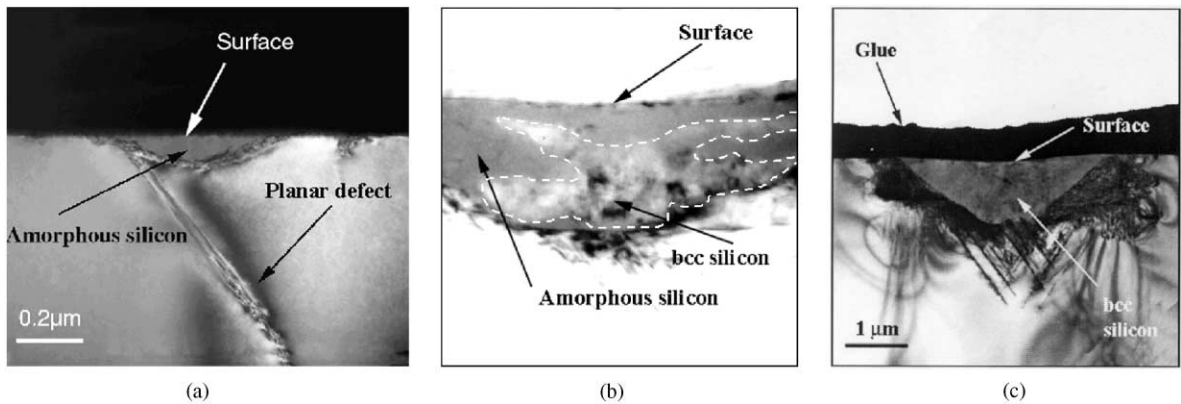


Fig. 1. The cross-sectional view of indentation-induced deformation in the subsurfaces of silicon specimens after complete unloading. Note the bcc-silicon particles with lighter colors, highlighted by the dotted white curves in (b). (a) 30 mN, (b) 70 mN, and (c) 90 mN.

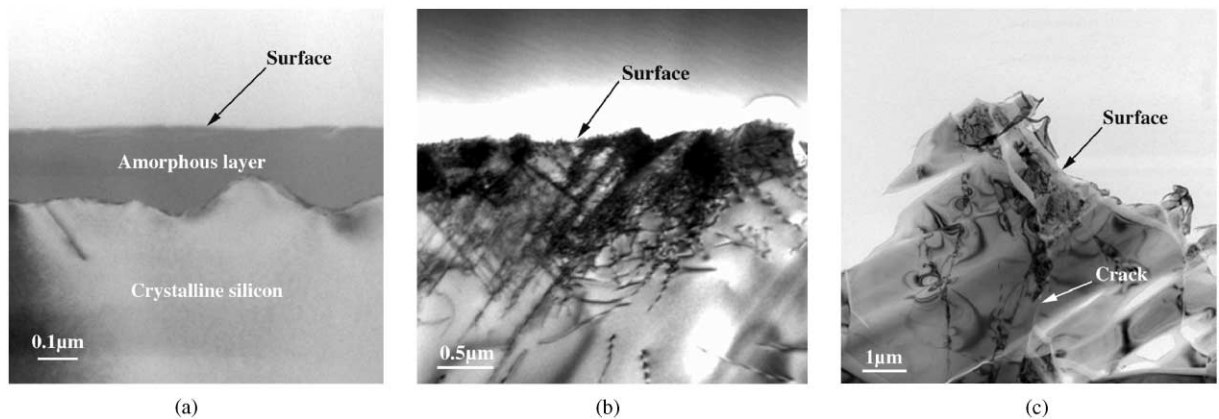


Fig. 2. A cross-sectional view of the scratching-induced deformation in the subsurfaces of the silicon specimens under various loads. Note the coexistence of dislocations and planar defects in (b). In both the cases shown in (b) and (c), amorphous layers on the scratched surfaces dislodged due to micro-cracking and can no longer be viewed on the photos. (a) 4.4 N, (b) 7.8 N, and (c) 11.2 N.

The indentation-induced transformation zone has a nearly hemispherical shape under lower loads (Fig. 1a), but approaches a cone when the load increases (Fig. 1c). A characteristic of the material's microstructure in the zone is its dependence on the indentation load. At a low load between 30 and 40 mN, the amorphous phase of silicon covers the whole transformation zone (Fig. 1a). However, when the load is beyond 50 mN, crystalline particles of bcc-silicon appear in the middle of the zone, as shown in Fig. 1b. The bcc phase grows quickly with the further increment of the load and occupies almost the whole transformation zone when the load reaches about 90 mN, see Fig. 1c. During the deformation, planar defects also appear beneath the transformation zone but dislocations never occur. When the indentation load reaches 100 mN, micro-cracking appears. No chemical reaction takes place.

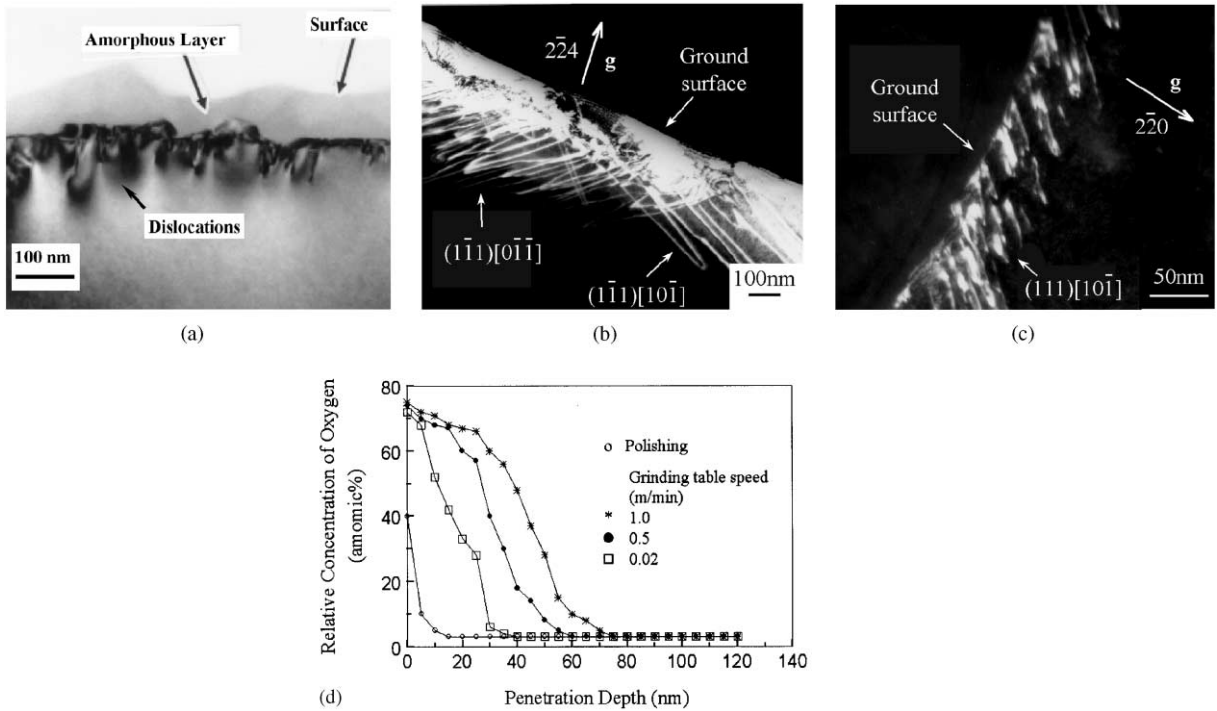


Fig. 3. Grinding-induced deformation and oxygen penetration accelerated by grinding and polishing: (a) subsurface deformation with both amorphous transformation and dislocations; (b) dislocations induced by grinding at a table speed of 1 m/min (note that two dislocation systems appear); (c) dislocations induced by grinding at a table speed of 0.02 m/min (note that only one dislocation system appears); (d) variation of depth of oxygen penetration with loading conditions.

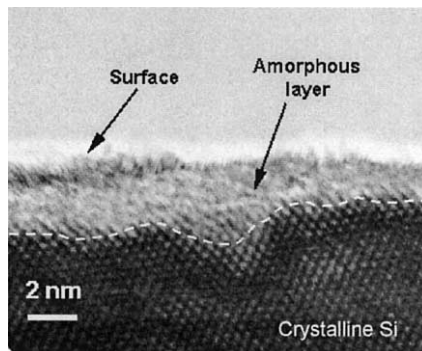


Fig. 4. A cross-sectional view of the polishing-induced subsurface deformation. Note that dislocations and planar defects do not appear. Amorphous phase transformation with oxidation is the only cause of plastic deformation.

The deformation caused by the single-point scratching possesses similar characteristics, i.e., amorphous phase transformation (Fig. 2a) constitutes the majority of the plastic deformation with intensive planar defects (Fig. 2b) when the normal scratching load is small. Although bcc phase does not emerge, dislocations do appear in this case, as shown in Figs. 2b and c. At a

greater load, e.g., 7.8 N, dislocations and planar defects are generated (Fig. 2b). Upon a further increment of the load, the material exhibits its brittle nature, demonstrated by extensive cracking and dislodging, see Fig. 2c. Similarly, no chemical reaction occurs.

As shown in Fig. 3a, grinding introduces significant amorphous phase transformation but at the same time initiates two systems of dislocations, as presented in Figs. 3b and c. In this case, the major deformation is still due to the phase change because only two slip systems of dislocations cannot form considerable macroscopic plastic deformation. Nevertheless, a chemical composition analysis of the ground components reveals that grinding accelerates chemical reaction through the application of the water-based coolant, Syntilo3. Oxygen penetrates into the deep subsurface of the silicon specimen, whose depth depends on the grinding conditions, as shown in Fig. 3d, and forms silicon oxide. Neither planar defects nor the bcc phase of silicon appear.

Unlike grinding and scratching that are two-body abrasions, polishing is a three-body abrasion. With the water-based α -Al₂O₃ slurry under a polishing load less than 450 mN, the scale of the abrasive indentation depths was at the nanometer level. Fig. 4, where each dot indicates a silicon atom, shows that the amorphous layer generated by polishing, which is above the dotted white curve, is very thin but its average thickness varies with the polishing load. Similar to grinding, penetration of oxygen and thus silicon oxidation in the subsurface has been considerable (see the curve in Fig. 3d) and the penetration depth increases with the load. An important phenomenon associated with polishing is that dislocations, bcc phase, and planar defects never appear.

3.2. Deformation mechanisms

Using the molecular dynamics analysis, Zhang and coworkers [17–20] found that under indentation, scratching, grinding or polishing, the regime of pure elastic deformation in silicon is very small. In a high vacuum environment, plastic yielding via amorphous phase transformation upon loading takes place first and then the transformation is caused by the distortion of the atomic lattice of silicon. The first transformation is governed by the octahedral shear stress, τ_{oct} , with its threshold $\tau_{\text{oct}} = 4.6$ GPa in $\langle 100 \rangle$ direction and $\tau_{\text{oct}} = 7.6$ GPa in $\langle 110 \rangle$ direction. However, the formation of both the amorphous and bcc phases after indentation depends on loading history. The amorphous phase appeared during loading will disappear upon unloading, if the unloading takes place at a small indentation load, because in this case a re-crystallization from the amorphous to the original diamond structure will occur during unloading. Only when the hydrostatic stress in loading, σ_0 , reaches a critical value, 8 GPa, such that a second phase transformation from amorphous silicon to β -silicon takes place, will an amorphous phase remain after unloading. Similarly, bcc silicon during unloading will not appear unless the maximum hydrostatic stress during loading is beyond 15 GPa. All these are consistent with the experimental observations shown in Fig. 1 and they indicate that the nature of the plastic deformation in silicon relies greatly on stressing history. Fig. 5 summarizes the above conclusions graphically.

Indentation promotes planar defects such as stacking faults, emanating from the bottom of the transformation zone. Their penetration depth changes with the indentation load, which increases until the load reaches about 90 mN and then decreases when the load increases further, as shown in Fig. 6. The phenomenon seems related to the initiation of micro-cracking at the intersections of the planar defects, i.e., the relaxation due to micro-cracking seems to hinder the

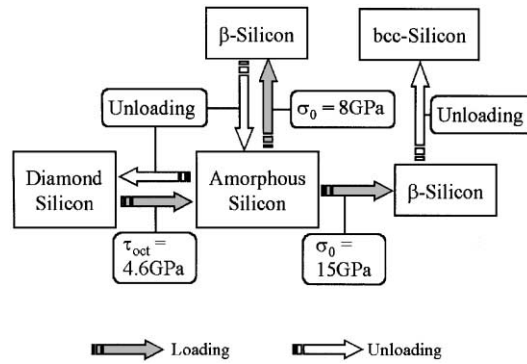


Fig. 5. Phase transformation mechanisms of silicon under indentation.

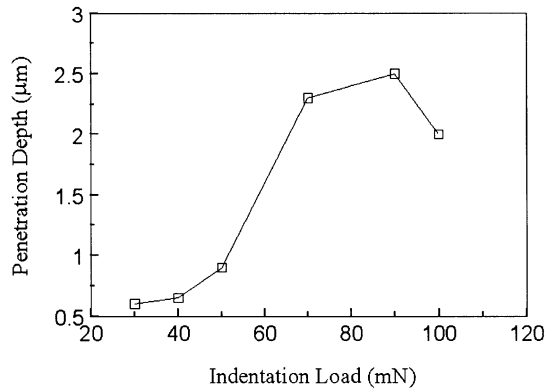


Fig. 6. The variation of penetration depth of the planar defects with indentation load.

planar defects from further penetration. No dislocation before micro-cracking means that planar defects are easier to be initiated in silicon at ambient temperature.

In scratching, planar defects still dominate at lower scratching loads but dislocations develop quickly as the load increases. Using a triangular model of moving heat source based on the theory of heat conduction [21], it is found that the maximum temperature rise in silicon under scratching is 150°C at the load of 4.4 N but reaches 200°C and 340°C at 7.8 and 11.2 N, respectively. Hence, temperature rise seems to play a key role in activating dislocations.

The grinding operations bring about dislocations only beneath the transformation zone, as shown above in Figs. 3a–c, without planar defects. In all the grinding cases, the maximum temperature rise in the silicon subsurfaces exceeds 200°C . Thus, compared with the conditions in indentation and scratching, it is clear that at high temperatures, dislocations are easier to be activated in silicon.

On the other hand, polishing never introduces dislocations, planar defects or micro-cracks. Compared with all the other operations above, a characteristic in polishing is that the abrasive penetration into a silicon specimen is on the order of a nanometer. This indicates that at the

nanometer scale, deformation via amorphous phase transformation is a more favorable mechanism. The theoretical results from molecular dynamics analysis presented above do support the conclusion.

3.3. Onset of amorphous transformation

The deformation of silicon can be divided into two stages, elastic deformation and plastic deformation. The plastic deformation, using the generalized definition in this study, is caused by all the irreversible atomic movements, such as the phase transformations, and the invasion of foreign atoms, such as the chemical reaction revealed in the experiments. Thus, to describe the initial yielding in silicon, three groups of factors must be considered. The first is the stress state that is, the stimulus of the atomic motion. The second is the mechanical property of silicon, determined by the strength of atomic bonds and lattice structure, which governs the response of the atoms to an external stimulus. The third is silicon's chemical property that specifies the extent of possible chemical reactions under complex loading conditions. Macroscopically, it is reasonable to assume that at a point in silicon when a certain combination of the principal stresses and the parameters representing the mechanical and chemical properties reaches a critical status, an irreversible deformation occurs at this point. Mathematically, it can be written as

$$F(\sigma_1, \sigma_2, \sigma_3; k_1, k_2, \dots, k_m) = 0, \quad (1)$$

where F is a function to be determined and k_i ($i = 1, \dots, m$) are the necessary material variables that describe all the possible irreversible deformations in silicon, including phase transformations, dislocations, planar defects, and chemical reactions. It is easy to imagine that a complete expression of F must be very complex. However, both the experimental investigation and molecular dynamics analysis demonstrate that deformations caused by dislocations and planar defects under the loading conditions in this study are minor compared with that due to phase transformation. On the other hand, it is still unclear to which extent the chemical reaction influences initial yielding. Thus in the first trial below, only the amorphous phase transformation will be considered.

The above experimental observations indicate that hydrostatic stress in loading may play a role in the phase transformation. Thus when ignoring the atomic lattice anisotropy of silicon, Eq. (1) may be taken as

$$F \cong \alpha I_1 + (1 - \alpha) \sqrt{J_2} - k = 0, \quad (2)$$

where I_1 is the first stress invariant, J_2 is the second invariant of deviatoric stress tensor and k and α ($0 \leq \alpha \leq 1$) are material constants to be determined. Clearly, α is an indicator of the contribution of hydrostatic stress, with $\alpha = 0$ representing its zero contribution and $\alpha = 1$ showing its full contribution. By fitting the indentation and scratching results obtained above, it is found that α is nearly zero. This indicates that in the case without chemical reaction, octahedral shearing governs the initiation of the amorphous phase transformation at loading. The effect of hydrostatic stress on the initial transformation from diamond to amorphous structure is trivial.

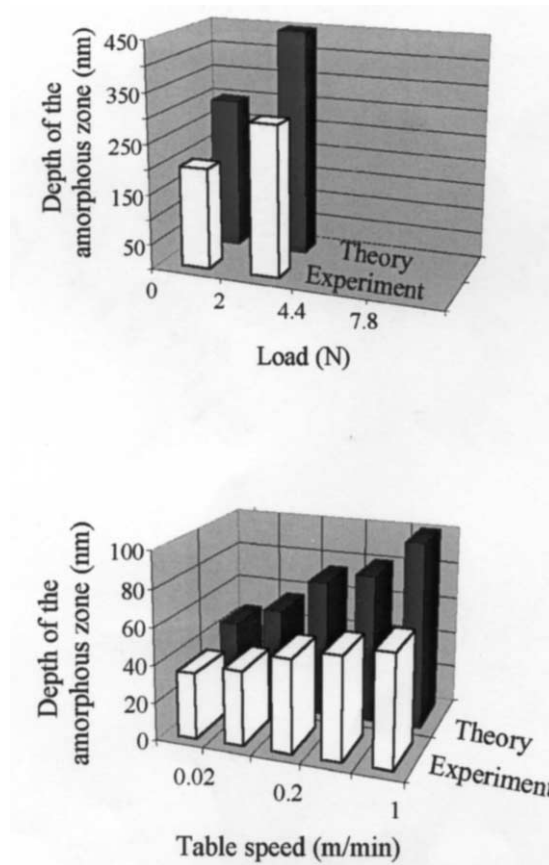


Fig. 7. Thickness of amorphous layer: experimentally measured (after complete unloading) and theoretically predicted (before unloading): (a) in scratched specimens; (b) in ground specimens.

Criterion (2) predicts very well the initiation of the amorphous phase induced by indentation, scratching, and grinding. For example, Fig. 7 compares the experimentally measured thickness of the residual amorphous layer with the predictions of the criterion. It is reasonable that the theoretically predicted thickness is greater because the experimental measurement was performed after unloading and according to the study of molecular dynamics simulation [17], a re-crystallization from amorphous to diamond silicon occurs at unloading, which makes the residual thickness of amorphous silicon much smaller compared to that before unloading. In calculating the stress field in a specimen subjected to scratching and grinding, the method developed by Zarudi and Zhang [12] was employed.

However, the criterion does not predict well the amorphous change introduced by polishing. An experimental examination shows that polishing under a normal load of 147 mN results in an amorphous layer of 1.3 nm in thickness. Nevertheless, the application of criterion (2) concludes that such an amorphous layer can be generated only when the effective modulus of the polishing

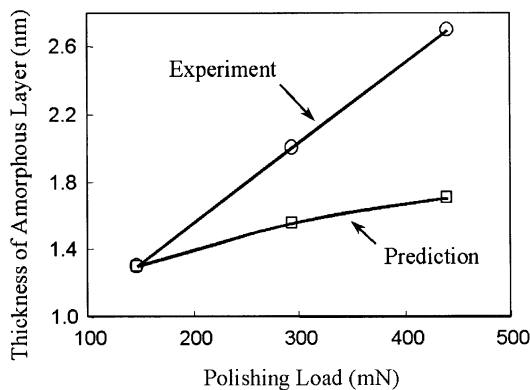


Fig. 8. A comparison of the theoretical prediction of amorphous layer thickness with the experimental measurement. The loading is via polishing with the water-based α - Al_2O_3 slurry. $E^* = 5$ MPa is used in the theoretical calculations.

system, E^* , is 5 Mpa, where

$$E^* = \left(\frac{1 - \nu_s^2}{E_s} + \frac{1 - \nu_p^2}{E_p} \right)^{-1}$$

in which E is Young's modulus, ν is Poisson's ratio, subscript "s" indicates silicon and subscript "p" stands for the abrasive-polishing pad.¹ However, when this E^* value is used to calculate the thickness of amorphous layer under 294 and 441 mN, it leads to 1.55 and 1.7 nm, respectively, while the experimental results under these loads were 2 and 2.7 nm. This means that the criterion significantly underestimates the extent of amorphous change and that the actual phase transformation is a much stronger function of the polishing load than the criterion's prediction, as shown in Fig. 8.

There are several factors that may cause the above inconsistency. First, if the amorphous change is mainly introduced by the mechanical loading, then the only explanation to the mismatching is that the effective modulus of the polishing system, E^* , must have varied sensitively with the polishing load. Unfortunately, an experimental measurement showed that E^* in polishing was almost a constant. This means that there exist other mechanisms affecting the amorphous initiation. By recalling the penetration of oxygen and the change of atomic bonding (i.e., the formation of silicon oxide) in the amorphous zone during polishing, as reported in the previous section, a possible cause of the above discrepancy between the theoretical prediction, which only considers the mechanical stress induced phase change, and the experimental observation, which is the result of the coupled effect of the mechanical loading and chemical reaction, is the invasion of the foreign atoms, oxygen atoms in this case. The invasion brings about the chemical reaction and may have reduced the threshold of octahedral shear stress for the initial transformation of the amorphous phase. A further investigation on this issue is currently in progress at the authors' research laboratory.

¹ Here the abrasive-pad and silicon specimen in polishing are considered to be the counterparts of a contact system.

4. Conclusions

The present investigation has led to the following major understandings:

- (1) The mechanism of plastic deformation in mono-crystalline silicon subjected to complex loading is very complicated, involving multi-phase transformations, dislocations, development of planar defects, and chemical reactions.
- (2) In the high vacuum environment, the octahedral shear stress enables the initiation of the phase transformation in silicon from its diamond structure to amorphous structure and the effect of the hydrostatic stress is trivial. However, the hydrostatic stress plays a key role in the further development of the transformed material, including the subsequent phase changes to β -silicon and bcc-silicon.
- (3) Chemical reaction may have reduced the threshold stress for the initiation of amorphous phase transformation. A further study is necessary to understand the principle.
- (4) There exists an unconventional elastic deformation regime in silicon, within which the amorphous phase formed during loading will disappear upon unloading because of a reverse transformation. The boundary of this regime is the formation of β -silicon during loading.

Acknowledgements

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