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Plastic deformation of silicon during contact sliding at ambient temperature

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Evidence of plastic deformation during contact sliding of silicon under relatively low loads at room temperature is presented. Sapphire spheres were slid against Si (1 0 0) under various normal loads at temperatures above and below the critical temperature. Upon chemical etching, pits that are attributed to dislocations developed along the sliding track for all experiments. This suggests that plastic deformation can readily take place in covalent solids, such as silicon, even at temperatures far below the critical temperature. The results of this work support the view that frictional force and energy dissipation are largely caused by plastic deformation of the materials near the sliding contact even under relatively low loads.

1. Introduction

The basic understanding of the frictional interaction is that when two solids come into contact under a certain load, a complex mesh of asperity interlocking is created at the contact interface. Consequently, when sufficient shear stress is applied, permanent deformation of the interfering asperities must occur in order to allow relative motion. The frictional work must be done to cause such deformation. In the case of crystalline solids, plastic deformation due to dislocation generation and motion is the dominant mode by which permanent deformation takes place, allowing the relative motion of asperities. Once wear particles are created as a result of plastic flow, the frictional force increases further due to the ploughing action by these wear particles. Eventually a steady state is reached when the ploughing by wear particles reaches an equilibrium state. The mechanical effects are augmented or reinforced by adhesion effects. These mechanisms of friction are well supported by experimental results which show signs of severe plastic flow of metals as a consequence of interactions at the sliding interface [1, 2].

In the case of covalent solids, however, it has been reported that owing to the directional characteristic of the bond type, twisting of the bonds is necessary for dislocation migration, which cannot occur at temperatures below a certain critical level [3]. At relatively low temperatures such solids are commonly known to undergo elastic-fracture behaviour, and therefore, do not experience plastic deformation. To support this view, researchers conducted indentation tests on silicon and germanium after which the specimens were etched to reveal dislocations. In the case of indentations performed below the critical temperature level (500–550 °C for silicon and 200–250 °C for germanium), etch pits did not appear, which suggested that dislocations did not migrate beyond the immediate vicinity of the indentation. However, for indentations performed above the critical temperatures, dislocation etch pits were observed beyond the area of indentation upon etching.

On the other hand, some researchers have proposed that plastic deformation can readily take place in covalent solids even at low temperatures if the stresses are high enough. Eremenko and Nikitenko [4] have reported that covalent solids such as silicon and germanium can deform plastically at low temperatures if stresses are comparable to those predicted for strengths of ideal solids. Gilman [5, 6] suggested that at low temperatures, dislocations can escape from their pinned positions by a stress-activated tunneling process rather than by thermal activation, in which case the stresses need not be as high as those predicted by Eremenko and Nikitenko. Quite recently, Shin *et al.* [7] showed that during indentation of silicon, plastic flow preceded cracking behaviour at ambient temperature.

This paper addresses the mechanism of material flow during contact sliding of a single-crystal covalent solid, namely silicon. This work demonstrates that plastic deformation is responsible for the frictional energy dissipation during contact sliding even for covalent solids that are generally known to favour deformation by fracture rather than by plastic flow at temperatures below the critical temperature.

2. Verification of plastic deformation by etch pits

2.1. Previous work

In previous work conducted to identify the microscopic mechanisms of friction, friction tests were performed

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at room temperature where single-crystal silicon specimens were slid against a sapphire sphere under loads below 5 gf (1 gf = 9.8067×10^{-3} N) [8]. The approach was to characterize the specimens with respect to surface damage following the friction experiments, and furthermore to assess the mechanisms involved in generating the scar, or the frictional force. It was discovered that under loads of about 3 gf, absolutely no sign of damage was apparent even when the surface was characterized using scanning electron microscopy as well as a high-resolution surface profilometer. However, upon chemically etching the silicon specimens, atomic-scale damage pits, that were attributable to dislocations, developed along the sliding track. This result subsequently led to the conclusion that frictional interaction between two solids in contact sliding is a destructive process where plastic deformation is the primary cause in the case of crystalline solids [9]. The work presented here was undertaken to verify if the pits were indeed due to dislocations generated during sliding.

2.2. Experiments under various normal loads and temperatures

The most direct method of verification is to etch the surface where definite signs of plastic deformation exist and to compare the etch pits with the ones found in the previous work. As mentioned in the previous section, no sign of surface damage could be observed directly for contact sliding tests conducted with normal loads below 3 gf. Therefore, sliding tests were conducted at higher loads where surface damage could be seen directly without etching. All sliding tests were done on Si (1 0 0) using a 3 mm diameter sapphire sphere as the pin. The surfaces were examined by the Nomarski interference contrast method before and after etching in 10 wt % KOH solution at 70 °C for 60 s.

Fig. 1 shows the damage along the sliding path for the case of 20 gf normal load sliding. Dispersed streaks of grooves are formed as a result of plastic flow caused during contact sliding. Another specimen, which was slid under the same conditions and showed similar signs of surface damage, was etched to reveal the dislocations generated during plastic deformation. Etch pits are developed along a much wider path, which indicates that the actual "affected" zone extends beyond the obvious boundaries of the sliding path (Fig. 1b). The surface morphology generated upon chemical etching with KOH is shown in Fig. 1b. This indicates that etch pits are caused by dislocations generated during plastic deformation.

Under a much higher load of 100 gf, signs of fracture appeared in addition to the grooves along the sliding path (Fig. 2a). The curved fracture lines, or semicircular cone-cracks, are commonly observed with brittle materials [10]. Another specimen was etched to reveal the dislocation etch pits (Fig. 2b). In certain areas, deep pits were observed which seem to occur near the fractured regions. The results of the low and high normal load experiments show that plastic deformation precedes the fracture regime even at room temperature.

The effect of temperature on plastic flow and etching was also investigated. Covalent solids are commonly known to flow plastically at high temperatures, and therefore, more dislocations are expected to be generated at higher temperatures during contact sliding under the same load. Sliding tests were conducted at two temperatures: 25 and ~ 500 °C. The normal loads were 1 and 10 gf. Fig. 3 shows that the dislocation etch pits are larger, though fewer in number, for the specimen slid at high temperature. Note that the etch mark which appears to be continuous in Fig. 1a actually consists of small pits similar to those observed previously. Under 10 gf normal load, the etch pit density is greater as expected and deep pits indicative of severe plastic flow can be observed (Fig. 4). The morphology of the etch pits is similar for specimens tested at low and high temperatures and more importantly, all the etch pits are identical in appearance to those shown in Fig. 1b. This shows that the etch pits are indeed caused by dislocations generated due to plastic deformation.

The mechanisms for the etch-pit formation are extremely complicated. In the case of etch pits developed as a result of contact sliding action under a spherical indentor, the pits are relatively ill-defined compared to

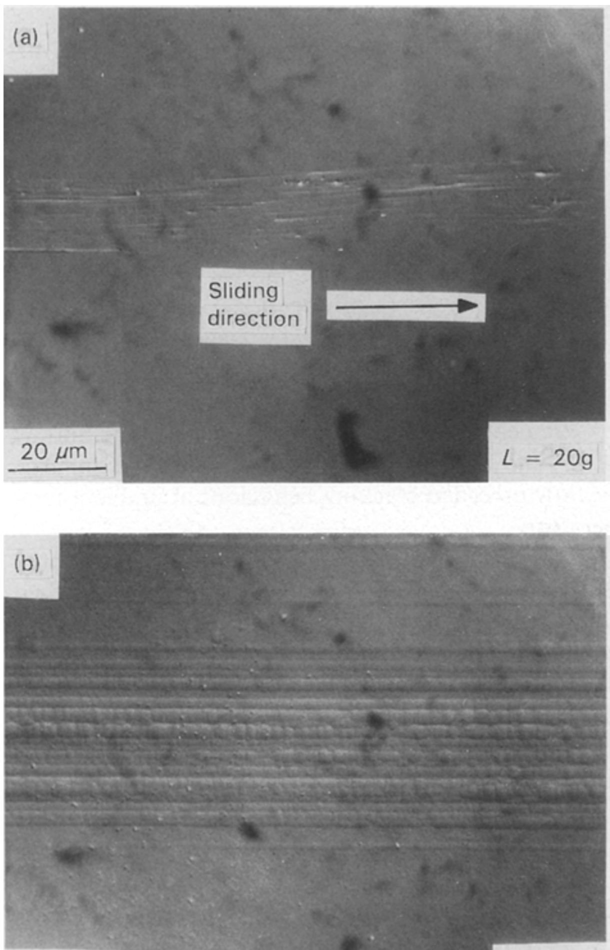

Figure 1 Nomarski interference contrast micrographs of Si (1 0 0) surface slid against 3 mm diameter sapphire pin under 20 g load: (a) unetched and (b) etched.

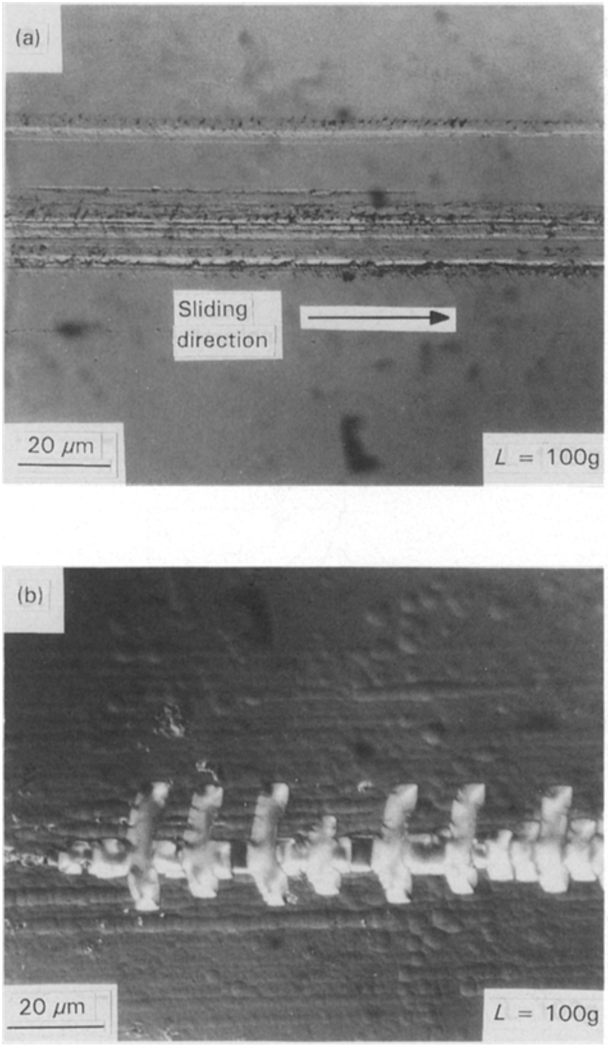

Figure 2 Nomarski interference contrast micrographs of Si (1 0 0) surface slid against 3 mm diameter sapphire pin under 100 g load: (a) unetched and (b) etched.

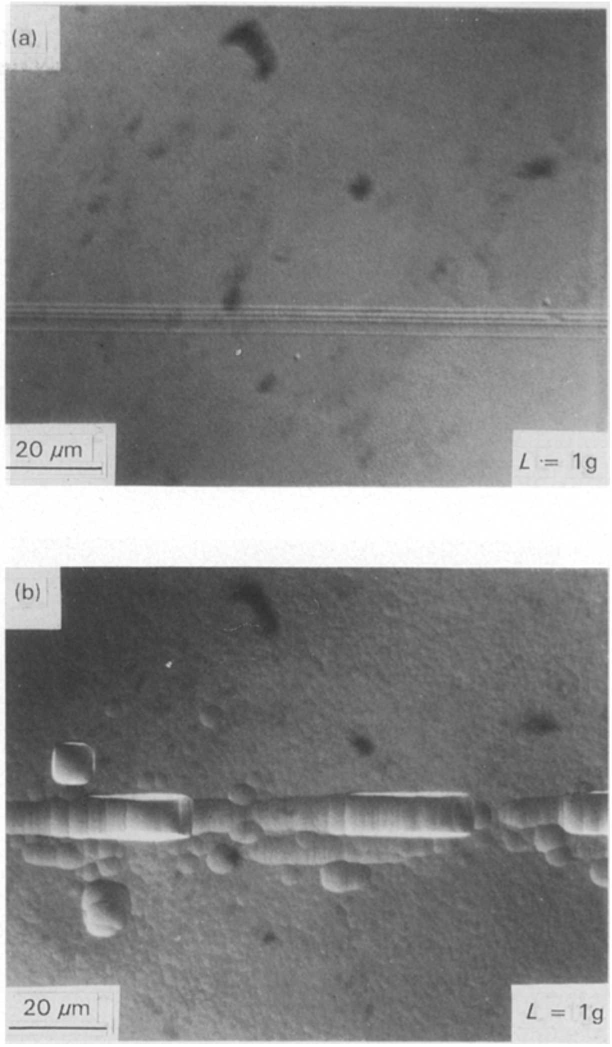

Figure 3 Nomarski interference contrast micrographs of etched Si (1 0 0) surface slid against 3 mm diameter sapphire pin under 1 g load at (a) $T = 25\,^{\circ}\mathrm{C}$ and (b) $\sim 500\,^{\circ}\mathrm{C}$.

dislocation etch pits found on ionic solids such as LiF and MgO, following indentation tests and etching [11]. It has been well established in the literature that dislocations will be etched preferentially due to their relatively high-energy states. However, not all dislocations are necessarily etched, and therefore, one-to-one correlation between etch-pit density and dislocation density is not expected. Also, other types of defects, such as stacking faults, impurities, and vacancies, may lead to etch pits, although the etch-pit shapes are not expected to be the same [12]. Although, quantitative assessment of plastic deformation through etch pits is extremely difficult, the etch pits developed along the sliding path confirm the importance of microscopic plastic deformation in generating frictional force at room temperature, even for covalent solids.

3. Critical stress for plastic deformation

The stress required for plastic deformation at low temperatures can be roughly estimated based on Gilman's approach [5, 6]. The rate of dislocation migration in the case of a thermally activated process is proportional to $\exp[-(\varepsilon - \sigma V)/k_B T]$ where ε is the dislocation binding energy, σ is the local stress, k_B is Boltzmann's constant, T is the absolute temperature, and V is the atomic volume. For the tunnelling process, the rate is proportional to $\exp(-\varepsilon/2\sigma V)$. The inequality expression for local stress level beyond which the tunnelling process dominates is given by [6]

$$\sigma > \left(\frac{\varepsilon}{4V}\right)\left\{1 \pm \left[1 - \left(\frac{4k_B T}{\varepsilon}\right)\right]^{1/2}\right\} \qquad (1)$$

For $4k_B T \ll \varepsilon$, the above inequality reduces to

$$\sigma > \frac{\varepsilon}{4V} \qquad (2)$$

and the transition temperature, T_t, for $2\sigma V \ll \varepsilon$ is given by

$$T_t \approx \frac{2\sigma V}{k_B} \qquad (3)$$

For a contact asperity volume of about $(0.5\ \mathrm{nm})^3$ and at 300 K, in order to induce dislocation migration the local stress should be greater than about 17 MPa. This derivation does not contain enough material parameters to discriminate between various covalent solids whose flow stresses can be quite different. It nevertheless gives a rough indication of what stresses to expect

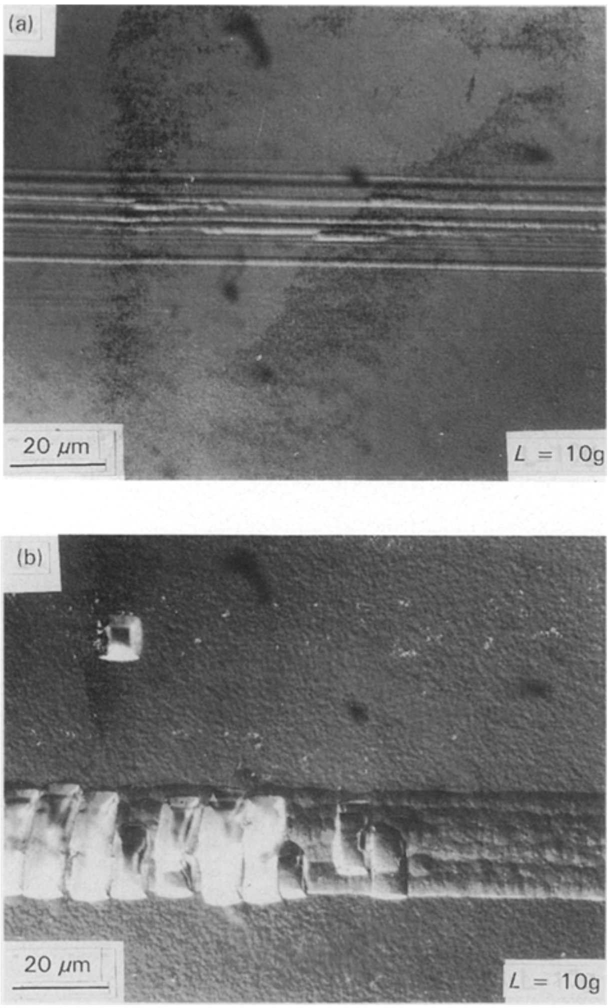

Figure 4 Nomarski interference contrast micrographs of etched Si (1 0 0) surface slid against 3 mm diameter sapphire pin under 10 g load at (a) $T = 25\,°C$ and (b) $\sim 500\,°C$.

for plastic deformation of covalent solids at low temperatures. A critical stress of 4.5 MPa has been reported for dislocation mobilization at about 600 °C in the case of Czochralski grown silicon with a phosphorus atom concentration of $6.2 \times 10^{18}\,cm^{-3}$ [13]. At room temperature the critical stress is expected to be significantly higher.

Owing to the anisotropic behaviour, the critical stress of a single crystal depends on the crystal structure and the direction of the applied stress. Plastic deformation by slip will occur if the shear stress on the slip system exceeds the critical shear strength. The critical shear strength along the slip plane for an Si (0 0 1) indented in the $\langle 0\,0\,\bar{1} \rangle$ direction can be estimated, using the schematic illustration of the problem shown in Fig. 5a.

For silicon, which has a diamond cubic structure, the closest-packed planes are $\{1\,1\,0\}$ and the shortest translation direction is $\langle 1\,\bar{1}\,1 \rangle$ [14]. The $\{1\,1\,1\}$ $\langle 1\,\bar{1}\,0 \rangle$ slip system is also a common slip system for diamond-structured crystals. The slip plane and the glide direction are identified in Fig. 5b. Note that the Schmid factor, m, which relates the resolved shear stress to the applied stress is (cos45°) (cos54.7°), or 0.41 for the $\{1\,1\,0\}\langle 1\,\bar{1}\,1 \rangle$ slip systems. For the $\{1\,1\,1\}\langle 1\,\bar{1}\,0 \rangle$ slip system, the Schmid factor is 0.58.

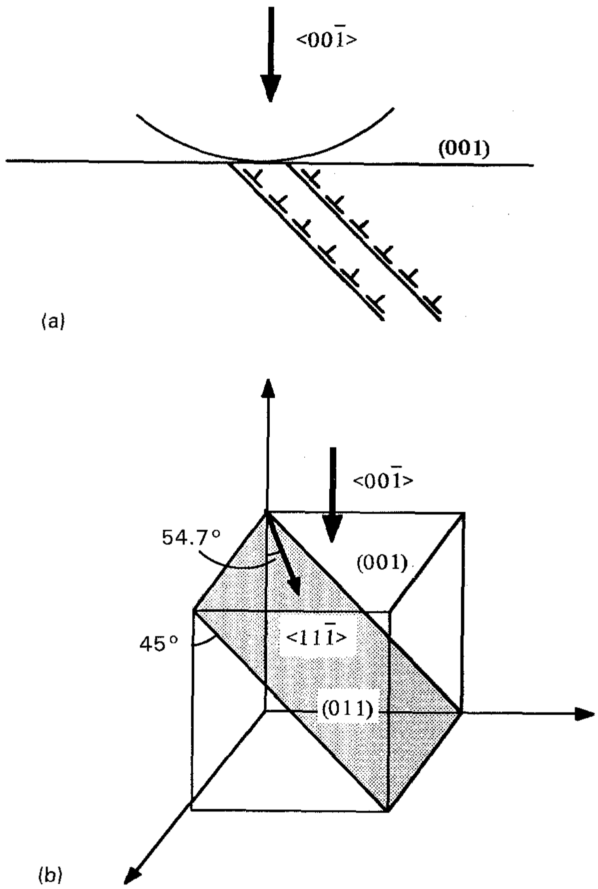

Figure 5 (a) Schematic illustration of slip when indented in the $\langle 0\,0\,\bar{1} \rangle$ direction on Si (0 0 1); (b) primary slip system for an infinitesimal element under the indentor.

Gilman [15] observed that indentation hardness, H, of covalent crystals can be related to the glide plane shear modulus, G_{gp}, by

$$H = 0.167\, G_{gp} \tag{4}$$

For cubic crystals

$$G_{gp} = 3[S_{44} + 4(S_{11} - S_{12})]^{-1} \tag{5}$$

where S_{11}, S_{12}, and S_{44} are the compliances (for silicon $S_{11} = 8\,MPa^{-1}$, $S_{12} = -2.2\,MPa^{-1}$, and $S_{44} = 12\,MPa^{-1}$ [16]). Also, from maximum shear stress below the indentor derived from elasticity theory, the following relationship between the maximum shear strength, τ_m, and the hardness is given as [15]

$$\tau_m = 0.33\, H \tag{6}$$

Then, from Equations 4 and 6

$$\tau_m = 0.055\, G_{gp} \tag{7}$$

For $G_{gp} \simeq 58$ GPa, $\tau_m = 3.2$ GPa. Finally, for the indentation geometry considered here, the resolved critical shear stress, τ_{rss}, can be obtained through the previously obtained Schmid factor of 0.41 as

$$\tau_{rss} = m\tau_m \simeq 1.3\,\text{GPa} \tag{8}$$

Thus, during contact, microplastic deformation is expected to occur along $\{1\,1\,0\}\langle 1\,\bar{1}\,1 \rangle$ slip system for shear stresses exceeding 1.3 GPa. Though this stress may seem to be too high for indentations using spherical indentors, the contact stresses under the micro-asperities can easily exceed this stress level even under loads as small as a fraction of a gram.

Resolving shear stress along the slip planes for contact sliding systems is much more complicated due to the multiple slip systems simultaneously affecting the deformation behaviour [17]. Kulhmann-Wilsdorf [18] proposed that for steady-state sliding the friction coefficient should be equal to the Schmid factor in the subsurface region. Because the friction coefficient is a factor that relates the tangential force to the normal force, it is plausible that the geometry of the slip systems, represented by the Schmid factor, will play an important role in determining the friction coefficient value. However, the predominant factor is expected to be the actual geometry of the contact interface between the two solids in relative motion.

4. Conclusions

Plastic deformation is the primary mechanism for frictional energy dissipation during contact sliding of two covalent solids even under relatively low loads at ambient temperature. This is evident from dislocation etch pits detected from low-load contact sliding tests of single-crystal silicon specimens slid against a sapphire sphere. The etch pits observed with specimens tested at ambient temperatures were similar in morphology to those tested at temperatures above the critical as well as under high loads. Thus, even in the case of solids with directional bonding characteristics, mechanical interactions such as plastic deformation due to asperity deformation and ploughing cannot be ignored in modelling the frictional behaviour.

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