In situ observation of nano-abrasive wear

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Abstract

We have studied the nano-scale wear behavior using an in situ nanoindentation device in a transmission electron microscope. Nano-scale sliding experiments were conducted under TEM. In order to trigger localized changes, we used a gold-coated diamond indenter to slide against single crystal silicon (100). With the movement of the indenter, cracks along the elastic strain contours in the silicon were observed. Energy analysis was carried over possible mechanisms. It was observed that the onset of abrasive wear was due to cracks formation along the contour. Analysis of the possible mechanisms that lead to the abrasive wear is presented. Observations are presented as initial work into experimentally observing the fundamental mechanisms.

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1. Introduction

Wear is one of the most important phenomenon that takes place at a material’s interface. The coexistence of ductile and brittle behavior in the subsurface region under sliding contact is yet to be fully understood. Conventional methodology reports both brittle fracture (surface cracks and subsurface voids) as well as plastic deformation [1–5]. In this research, we have directly observed the nano-scale wear using an in situ technique.

The development of nano-contact techniques has advanced our understanding of nano-mechanical and surface properties over the past few years [6]. Introduction of an atomic force microscope or a nano-indentater into the transmission electron microscope, opened more areas of investigation into the nano-mechanical properties [7–9]. The in situ nanoindentor consists of an indenter attached to a metal rod that is actuated by two mechanisms. For coarse positioning, the indenter can be moved in three dimensions by turning screws attached to a pivot at the end of the rod. For fine positioning, including the actual scratch, the indenter is moved in three dimensions with a piezoelectric ceramic crystal, which expands in response to an applied voltage. The tip’s sliding speed is controlled manually by controlling the voltage applied to the piezoceramic element, which leads to a load that is dependent on both the applied voltage on to the piezoceramic crystal and the movement during sliding.

2. Experiments and results

The nano-sliding experiments were conducted on a single crystal silicon (Si) (110). The indenter was made of diamond (Berkovitch geometry) coated with a gold film having a thickness less than 1 μm. The Mohr hardness of single crystal silicon is 7.0 [10] and that of gold is 2.5–3 [11]. The single crystal silicon was prepared using bulk silicon micromachining, leaving behind a thin wedge window that is electron transparent at the peak. The incident beam is a 30 kV Ga+ ion beam. The gold-coated indenter was mounted on a piezoceramic actuator, which both controlled its position in three dimensions and forced it into the edge of the sample. During in situ nano-sliding the deformation was observed and recorded in real time. Using either bright-field or dark-field imaging conditions, diffraction patterns were taken directly before and after sliding. The sliding speed of the indenter was about 14 nm/s. The indenter was kept stable so that the applied force was considered constant.

Fig. 1 shows a series of photos taken during the in situ nano-sliding experiment. Fig. 1a was taken right before the indenter
reached the Si surface. The lower left, dark area, is the indenter’s tip and on the upper right is the silicon surface. There are two thickness fringes shown. Fig. 1b was taken as soon as the indenter was in contact with the silicon. The straight lines (thickness fringes) have disappeared due to a slight change in the diffracting condition. However, some new fringes, circular in shape, appeared. The circular fringes are strain contours and represent the stress field directly. The contact area was on the edge of the left side of the photo (not completely shown). Fig. 1c was taken right after the first ring crack was formed. New stress rings appeared almost immediately. It was observed that it took about 5 s for the first onset to take place. Fig. 1d was taken after the indent moved further toward the right, and the contact point is clearly seen. Fig. 1e was taken right after the second ring (circular) crack was formed. It took about 27 s for the second onset to take place after the contact was engaged. The original fringes appeared again and the circular fringes were gone. Fig. 1f was taken after the indenter was removed away from the wafer. The circular shape remained and a piece of silicon wafer was attached to the indent.

Fig. 1 presents the initiation of wear. According to these initial results, we hypothesize three possibilities for wear initiation: formation and propagation of voids, fracture, or formation and connection of dislocations. In our results, the cracks observed
indicated brittle behavior during the initiation of wear. In the following, we compare the elastic energy needed for all three cases.

3. Energy comparison

To understand the relationship between the nucleation and propagation/growth of voids, fracture, and dislocations, we re-evaluated the energy dispersed during sliding. Firstly, we discuss the elastic strain energy needed to initiate a crack of length $2c$ [12]:

$$U_E = -\frac{\pi c^2 \sigma^2}{E}$$

where $\sigma$ is the tensile stress acting normal to the crack of length $2c$, and $E$ is the elastic modulus. $U_E$ is the amplitude (a few electron volts). More specifically, we assume that a crack is formed when a Si–Si bond is broken. The bond strength of gas Si–Si is $326 \pm 10.0$ kJ/mol, i.e., 3.38 eV. This will be the minimum energy needed to initiate a crack, without considering the compressive effects of surrounding atoms.

Second, we consider the total elastic energy per unit length of a screw dislocation. It can be formulated as

$$E_s = \frac{G b^2}{4\pi} \ln \frac{R}{r_0}$$

where $R$ and $r_0$ are the upper and lower limits of a screw dislocation, $G$ the shear modulus, and $b$ is the Burger’s vector. Similarly, for an edge dislocation, the elastic energy per unit length is

$$E_e = \frac{G b^2}{4\pi(1-\nu)} \ln \frac{R}{r_0}$$

where $\nu$ is the poison’s ratio. According to these two equations, if we consider $R$ to be comparable to the dislocation spacing and $r_0 \approx b$, then $R/r_0 \approx 10^3$. The dislocation energy is then about $5 \times 10^{-4}$ erg/cm, i.e., 8 eV per atom plane through which a dislocation passes.

Third, assuming there is a concentration gradient of gold or vacancies in silicon, there will be a flux of these through the silicon substrate. In equilibrium condition, the gold or vacancies will be distributed uniformly. The net flux $J_N$ of Au atoms is related to the gradient of the concentration $N$ by a phenomenological relation, i.e., Fick’s law:

$$J_N = -D \text{ grad } N$$

where $J_N$ is the number of atoms crossing unit area in unit time, $D$ the diffusion constant or diffusivity and has the units cm$^2$/s. The negative sign means that diffusion occurs away from regions of high concentration. The diffusion constant is formulated in the following equation:

$$D = D_0 \exp \left( \frac{-E}{k_B T} \right)$$

where $E$ is the activation energy for the diffusion process. The activation energy of Au diffusion in silicon is $E = 1.13$ eV and $D_0$ is $1 \times 10^{-3}$ cm$^2$/s. In order to diffuse, the gold atom must overcome the potential energy barrier. The diffusion of gold in silicon is considered to be in interstitial sites. Identical results apply to the diffusion of vacancies. If the potential energy barrier of $E$ is too high to overcome, the atom will only pass a fraction $\exp(-E/k_B T)$ of the time. Quantum tunneling through a barrier is usually important only for the lightest nuclei, because for a given energy, the de Broglie wavelength increases as the mass of the particle decreases.

For comparison, the formation of vacancies inside silicon is more energetically favorable than the formation of a crack or a dislocation or for the Au to diffuse into the silicon surface. In addition, there are three more factors promoting the formation of vacancies: shear or tensile stress, sufficient space for atoms to move around, and the motion of the indenter.

Fig. 2. Illustration of events taking place during sliding: voids, dislocations, and cracks.
How were materials detached from its surface during sliding? There are several detachment theories existing, including weakest spots due to shear [13], shear crack propagation [14], ploughing, wedge forming, or cutting [15]. Recent work by Schuh et al. in high-temperature nano-indentation experiments reported that the onset of dislocation plasticity was related to the nucleation of pre-existing vacancies [16]. In our work, it shows that the formation of vacancies within the near-surface region is thermodynamically possible.

Another complication for silicon is its many phases and transformations. Single crystal silicon has at least 12 crystal structures. Under stress, these structures undergo phase transformations [17–28]. These phase transformations occurred due to high pressure and volume change. Our experiments were conducted at the near-surface region. Therefore, the material has enough space to expand. As such, the volume change is considered less important in thereafter the phase transformation and is ignored.

As a summary, from the energy point of view, the formation of voids along the elastic stress contour initiates wear. These voids lead to fracture as well as dislocation formation under the sliding stress. The propagation of these voids is extremely localized. Once voids are formed, the further development of dislocations and fracture lead to material detachment—wear. To illustrate the wear process, a schematic explanation is shown in Fig. 2. According to the above analysis, the origin of wear is energetically favorable for the formation of voids leading to cracks.

4. Conclusions

In this work, we have demonstrated the ability to observe wear formation during in situ nano-sliding wear experiments. The subsequent occurrence of fracture and dislocation events lead to nano-scale wear. We foresee further development of the in situ observation of nano-scale wear that has not been possible until today.

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