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Secondary cracking at grain boundaries in silicon thin films

Jin Chen and Yu Qiao*

Department of Structural Engineering, University of California, San Diego, La Jolla, CA 92093-0085, USA

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In this article, we report irregular cleavage front transmission at grain boundaries in free-standing polysilicon thin films. When the orientations of two adjacent grains are correlated, the crack may bypass the boundary via a "tunneling" process. Similar behavior can also be achieved if the crack path curves in the grain-boundary-affected zone. Moreover, the separation of crack flanks can be aided by secondary cracking. These irregular modes of crack front behavior tend to lower the effective boundary toughness.

Keywords: Fracture; Thin films; Grain boundaries; Semiconductors

It has long been noticed that grain boundaries are important toughening elements in brittle polycrystalline materials [1–3]. Since the fracture resistance of crystallographic plane is constant, once a cleavage crack has propagated in a grain it will not stop unless the crack growth driving force decreases or the front encounters an obstacle, e.g. a grain boundary.

At a grain boundary, because the crystallographic orientations of the two grains across it are different, the cleavage cracking process is interrupted [4]. The crack must change its surface orientation so as to advance in the cleavage plane of relatively small surface free energy [5], i.e. the fracture surface becomes discontinuous. A certain portion of grain boundary must be separated to connect the cleavage facets, which demands additional fracture work [6]. According to a previous experiment on large iron–silicon bicrystals [7,8], the front would penetrate the boundary at locations where the local stress intensity is relatively high and/or the local fracture resistance is relatively low. The rest of the front is arrested by the persistent grain boundary islands (PGBIs) in between the break-through points (BTPs), until the PGBIs fail.

In a thin film material, in addition to the crystallographic orientation, the crack front behavior can also be affected by the film surfaces. As shown in Figure 1, when the film thickness is relatively small, there would be only a single BTP at a crack front. If there were multiple BTPs, the PGBIs would be too close to each other, causing a large crack-trapping effect [9]. Before the crack growth driving force can rise to a high enough level to overcome the barrier effect, the front would penetrate the boundary through the weakest BTP, i.e. other BTPs cannot be activated. Such a crack front transmission process will be referred to as the regular mode in the following discussion. Under this condition, the toughening effect of the boundary comes from the two PGBIs at both sides of the BTP. Through a weight-function analysis, an upper estimate of the boundary toughness, K_{GB}, can be given by [10]:

$$\frac{K_{GB}}{K_{sc}} = \left\{ \frac{w}{t} + \left(2.4 - 0.3\frac{t}{w}\right)^2 \left(1 - \frac{w}{t}\right) \right\}^{1/2} \times \sqrt{\cos\theta\cos\psi}, \quad (1)$$

where K_{sc} is the effective fracture toughness of the crystallographic plane, w is the width of the BTP area, t is the film thickness, and θ and ψ are twist and tilt misorientation angles, respectively.

While the regular mode was observed repeatedly, different crack front transmission behaviors can occasionally occur, as will be discussed in detail below. These "irregular" break-through modes take place when the crystallographic orientations satisfy certain requirements, and thus can be regarded as "extrinsic". In an irregular mode, the effective boundary toughness is lowered and the boundary may effectively govern the overall catastrophic failure. In order to evaluate the reliability of polycrystalline thin films, these behaviors must be taken into consideration.

The fracture experiment was conducted on a polysilicon heavily doped with boron. The average grain size

* Corresponding author. Tel.: +1 858 534 3388; e-mail: yqiao@ucsd.edu

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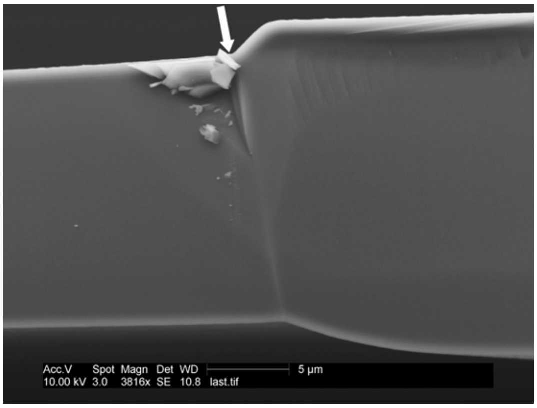

Figure 1. SEM fractography of regular mode of cleavage cracking across a grain boundary in a silicon thin film. The crack propagates from the left to the right. The arrow indicates the grain boundary.

was 5–10 mm. The as-received material was cut into wafers 4 mm thick and 200 mm in diameter. Silicon pieces 15×15 mm that contained through-thickness grain boundaries were harvested through electric spark cutting. They were further sliced to 0.2–0.3 mm thick films, mechanically ground to 80–100 µm, and finally chemically etched to 5–50 µm. The grain orientation was analyzed by Laue back reflection. The thin film sample was mounted in a compound-flexure microtesting machine [11]. A tensile load was applied at a constant rate of $10\ \mu m\ s^{-1}$ along the axial direction. When the critical condition was reached, the film would fail by cleavage cracking. The fracture surfaces were observed in an environmental scanning electron microscope (ESEM). The regular mode discussed above dominated the failure of most of grain boundaries [12]. The current study is focused on the irregular mode. Figures 2–4 show typically fractography.

Each silicon grain is a face-centered cubic (fcc) crystal. It can cleave along either {1 1 1} or {1 1 0} planes [13]; the crystallographic toughness of these planes is nearly identical. Due to the large number of possible cleavage planes, even when the crack propagates in a single crystal, the crack surface can shift among cleavage systems of similar orientations, and therefore the surface roughness is relatively high.

This characteristic of crack advance in silicon is essential to the phenomena demonstrated in Figures 2 and 3. In Figure 2, the crystallographic orientations of the two grains across the boundary are quite special. There are two sets of {1 1 1} planes having nearly the same orientation. While in ordinary materials such a coincidence is highly unlikely to occur, in a silicon thin film the possibility is considerably higher, because the $\langle 1\,1\,1 \rangle$ axis is the favorable growth direction [13], i.e. the crystal growth along $\langle 1\,1\,1 \rangle$ is faster than along other directions. As a result, during the deposition process, the crystal nuclei with other axes aligned with the film thickness direction would be buried, and most of the grains that can eventually become large, especially those of through-thickness grain boundaries, are of similar out-of-plane axes [14]. The major difference is the rotation angle. In Figure 2, in the grain behind the boundary ("A"), the crack advances along the (1 1 0) plane. If

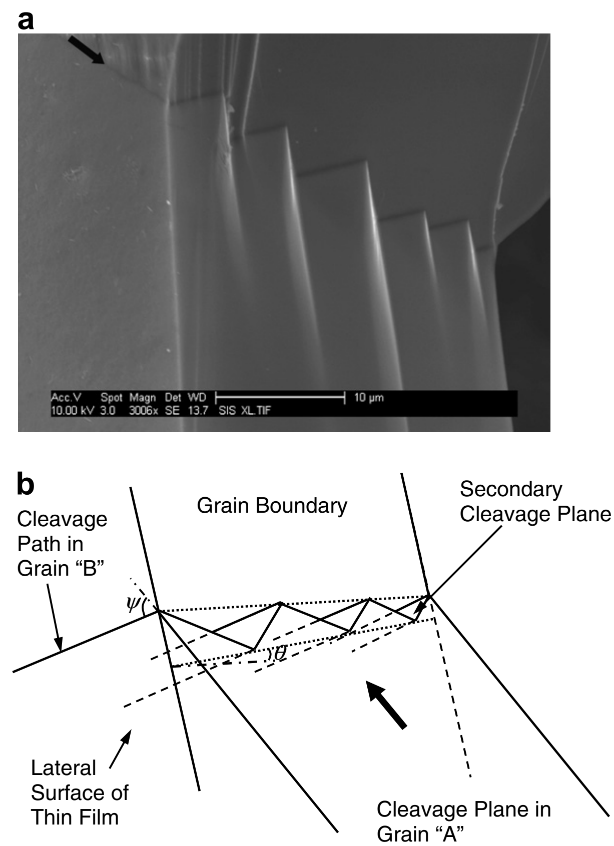

Figure 2. (a) SEM fractography and (b) schematic diagram of secondary cracking in the grain-boundary-affected zone. In the SEM image the crack propagates from the top to the bottom. The arrow indicates the grain boundary.

the crack front bypassed the boundary in regular mode and it kept propagating on a {1 1 0} plane in the grain ahead of the boundary ("B"), there would be relatively large misorientation angles between the fracture surfaces across the boundary. According to Eq. (1), if the BTP width, w, is taken as 2.5 µm, K_{GB}/K_{sc} would be around 15. Such a high boundary toughness makes the regular mode energetically unfavorable.

In Figure 2, it can be seen that about 1 µm away from the grain boundary, in grain "A", secondary cracking takes place along two {1 1 1} planes. Since the two grains are of correlated orientations, the {1 1 1} cleavage surfaces can extend smoothly to grain "B". In fact, from SEM fractography, little evidence of grain boundary interruption can be observed on the fracture surface. The grain boundary is more clearly shown at the lateral film surface. Because there is almost no additional grain boundary area that needs to be separated, the required crack growth driving force is quite small. The barrier effect mainly comes from the increase in fracture surface area, i.e. the increase in work of separation [15]:

$$\frac{K_{GB}}{K_A} = \sqrt{\cos\theta \cos\psi}, \qquad (2)$$

where K_{GB} is the effective stress intensity factor for the crack front to propagate through the grain boundary area, and K_A is the toughness of grain "A". In this case, K_{GB} is higher than K_A by only about 60%, much lower

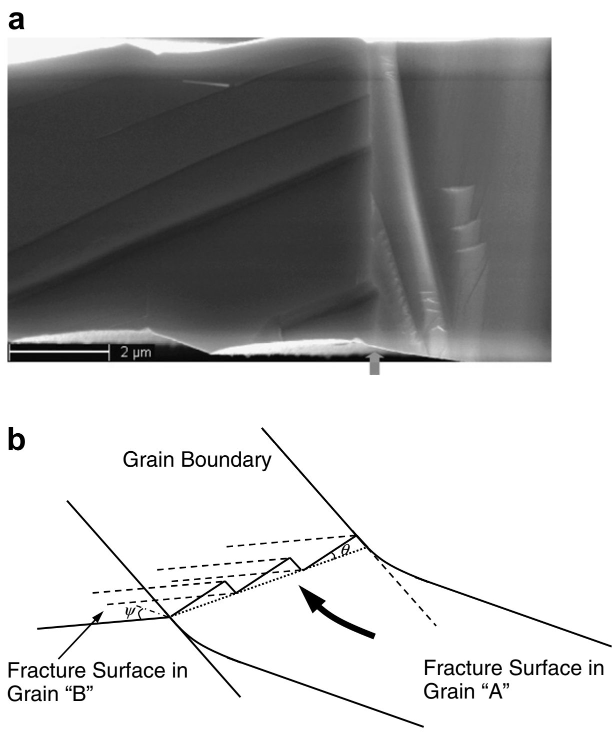

Figure 3. (a) SEM fractography and (b) schematic diagram of curving of cleavage path in the grain-boundary-affected zone. The crack propagates from the right to the left. The arrow indicates the grain boundary.

Figure 4. SEM fractography of cracking on multiple cleavage planes in the grain ahead of the boundary. The crack propagates from the left to the right.

than the toughness associated with the regular mode. The two {1 1 1} planes are not normal to each other and the crack front segments on them cannot propagate in parallel. Consequently, the secondary fracture facets rapidly merge together. The widths and the heights of the ridges formed by the facets keep decreasing. After the ridges extend into grain “B” by about 10 μm, they vanish, and the primary fracture surface becomes the {1 1 0} plane again. In this mode, the fracture surface “tunnels” through the grain boundary.

Figure 3 shows another irregular mode observed in the experiment. At first sight, it is somewhat similar to Figure 2. There are no clearly defined break-through windows. The crack front breaks down into a number of segments that directly advance on secondary fracture facets in grain “B”. However, the secondary facets do not vanish. The secondary facets extend deep into grain “B”, with similar river markings. The crystallographic orientations of grains “A” and “B” do not satisfy the requirement that the secondary facets are of the same orientation, i.e. the crack surface cannot directly change from the primary cleavage plane in grain “A” to the secondary planes in grain “B”. This crack front transmission mode is geometrically feasible due to the curving of crack path near the boundary in grain “A”. As depicted in Figure 3b, a few micrometers away from the boundary, the fracture surface is no longer flat. This characteristic length is compatible with the thickness of grain-boundary-affected zone (GBAZ) [16,17]. When the crack front approaches the boundary and enters the GBAZ, the crack-tip stress field is distorted. The anisotropy of grain “B” affects the crack front behavior. As a result, the fracture path deviates from the initial direction. If there were sufficient space, the fracture surface may shift to the secondary cleavage plane in grain “A” [18,19]. However, since the incident angle of the cleavage crack is relatively small, the fracture surface rapidly converges to the grain boundary plane. Once the grain boundary is exposed to the fracture surface, direct cracking into grain “B” along crystallographic planes becomes possible, as observed in Figure 3a. The fracture occurs along the most energetically favorable secondary planes, and the crack front breaks down into a number of segments propagating along different facets. In the sample shown in Figure 3a, if the crack front transmitted from the primary cleavage plane of grain “A” to that of grain “B” in regular mode, according to Eq. (1), the required crack growth driving force should be around $4 \times K_{sc}$. In the irregular mode, according to Eq. (2), the barrier effect of grain boundary is mainly caused by the increase in fracture surface area and the variation in fracture toughness is only around 70%.

Even when the crack front penetrates across the boundary in a similar way as occurs in the regular mode, secondary cracking can still take place in grain “B”. As shown in Figure 4, because the film is relatively thick, there are a number of BTPs. In regular mode, after the front penetrates into grain “B” at the BTPs, the grain boundary areas in between them need to be sheared apart so as to complete the fracture surface separation. In the sample under investigation, at the upper portion of the boundary the BTPs are close to each other, and thus the PGBI area is quite small. In the lower portion, however, the BTPs are far and few between, and shearing of these demands a high crack growth driving force. The variation in BTP distance is probably related to the kinetics of front transmission [7,8]. As the local stress intensity increases relatively slowly, the grain boundary is entirely exposed to the crack front, and BTPs can be fully developed. If the local stress intensity rises relatively rapidly, the optimum BTP structure may not be reached when local grain boundary starts to fail. As a result, the BTP distance can be large [20]. Once the front bypasses the boundary, no new BTPs can be formed. Under this condition, if the separation of the large PGBI is difficult, secondary cracking may take place if the secondary cleavage facets can connect the grain boundary and the primary cleavage planes. Since the secondary cleavage plane is of a relatively small twist misorientation angle with respect to the fracture surface in grain "A", the associated additional fracture resistance is relatively small. If the BTP distance is small, the PGBI can be separated even with a relatively low local stress intensity. Hence, secondary cracking cannot be activated, as shown in the upper portion of Figure 4.

To summarize, while the behavior of the majority of grain boundaries in silicon thin films are dominated by the regular mode, in which the boundary is sheared apart after the cleavage front penetrates at break-through points, secondary cracking can take place under a certain conditions. If the orientations of the two grains across a boundary are correlated, the cleavage crack can directly enter the grain ahead of the boundary via a "tunneling" effect. If the incident angle is relatively small, in the GBAZ the crack path can deviate and converge to the boundary plane, after which the crack can bypass the boundary without boundary separation. Even when crack penetration has taken place, if the BTP distance is relatively large, secondary cracking can occur in the grain ahead of the boundary so that the separated boundary area is reduced. In these irregular crack front transmission modes, the effective boundary toughness is lowered.

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