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Interaction of cleavage ridges with grain boundaries in polysilicon films

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ABSTRACT The morphology of cleavage surfaces across grain boundaries in free-standing silicon thin films was investigated. Three ridge–boundary interaction modes were identified. If the cleavage ridge was relatively deep, it could directly bypass a grain boundary. If it was relatively shallow, it would act as a stress concentrator promoting cleavage front transmission. It was also observed that due to the smoothening effect, the surface could become effectively less rough across a grain boundary.

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1 Introduction

When a cleavage crack propagates in a brittle polycrystal, grain boundaries are major obstacles [1, 2]. As the crack front approaches a grain boundary, due to the change in crystallographic orientation the stress field ahead of the crack tip can be distorted [3], and as a result in the grain boundary affected zone the effective crack growth driving force may decrease. When the front reaches the boundary, in order to advance onto the cleavage plane of the grain ahead of the boundary, the fracture surface must shift [4, 5]. Consequently, additional fracture work needs to be done to overcome the crack trapping effect of the grain boundary and to eventually separate it apart. The separated grain boundary areas distribute along the crack front quasi-periodically, with the characteristic distance ranging from 1–50 μm. In a thin film material, if the film thickness is smaller than this characteristic length, the space would be sufficient for only one breakthrough window [6–8].

As the crack propagates, river markings would be inevitably generated [9]. River marking is the terminology used to describe a ridge where two parallel cleavage facets meet. To connect the two planes, secondary cracking, often normal to the primary fracture surface, must take place. There are a variety of possible causes of river marking. For instance, when a crack front propagates across a relatively wide grain boundary, it must branch into a number of segments that advance on a series of parallel terrains [4]. The borders of the segments become river markings. Usually, it is assumed that the river markings are normal to the crack front, and therefore they indicate the local crack growth direction. In a single crystal, river markings can be formed as a crack front bypasses an inclusion, a secondary boundary, or a dislocation, so that the cleavage plane is sectioned. In the early years of metallurgy study, river markings were used to detect dislocations in clean crystals [10].

Since river markings commonly exist in cleavage cracking, it is of great interest to understand how they interact with grain boundaries. The current investigation is focused on fracture behaviors of polysilicon thin films, which has been an active area of research over the past two decades [11–16]. While a study on large samples may provide useful insights, the boundary conditions of a grain in the interior of a bulk material and a grain in a thin film with both of its sides exposed to free surfaces are different. In the interior of a bulk material the plane strain condition is dominant due to the confinement effect of surrounding materials. In order to obtain data that are immediately relevant to microelectromechanical systems, integrated circuits, etc., experiments must be performed on thin-film samples, which can lead to better processing and/or post-processing treatment techniques that enhance system reliability.

2 Experimental

The material investigated in the current study was polycrystalline silicon. The grain size was around 10 mm, and the wafer thickness was initially 4 mm. Since the grain size was larger than the thickness, most of the grains were through-thickness. It was heavily doped with boron so that its electric conductivity was sufficiently high for electrical discharge machining (EDM) [17].

In order to produce precracked samples for fracture experiments, partial quenching was performed. The wafer was first heated up to 450 °C and then partly immersed in cold water. The generated thermal cracks randomly stopped inside the wafer, many of which were arrested by grain boundaries. Those cracks were identified using an optical microscope and their tips were harvested by EDM cutting. Each pre-cracked piece was about 15 mm large, and was cut along the cross-sectional direction into 6–9 thick films. The thick films were thinned to 80–100 μm by mechanical grinding, and then to

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10–60 μm by wet etching. The etchant contained 7% hydrofluoric acid, 75% nitric acid, and 18% acetic acid, which is widely used in microfabrication [18]. The etching was performed in a teflon tank, and the etchant was circulated through a polypropylene pipe, driven by an Omega FPU-500 peristaltic pump at a constant rate of 30 ml/min. Prior to the mechanical grinding, the samples were surface treated for 5 days by chlorotrimethylsilane [19]. The pre-crack surfaces were coated by nonwettable silane groups, and thus were not affected by the etchant. By using a compound-flexure microtester [20, 21], the thin-film sample was pulled apart at a constant rate of 10 μm/s. The fractured samples were thermally cleaned in vacuum at 350 °C for 6 h, washed by acetone, and dried in air. They were observed in an environmental scanning electron microscope (ESEM), and those with large numbers of river markings were examined in detail. Note that the grain boundaries under investigation in the current study were not the ones that initially arrested the pre-crack tips. Typical results are shown in Figs. 1–4.

3 Results and discussion

Figure 1 shows that, if the cleavage surface in the first grain is relatively smooth, the crack front would penetrate across the boundary in the central part, where the local stress intensity is higher than the sections near the free surfaces [7]. The rest of the front is arrested by the boundary. As the crack growth driving force rises, the front penetrates deeper across the boundary. When the boundary is separated apart, its barrier effect is overcome and the front moves forward unstably. Across the boundary, the fracture surface changes its orientation to minimize the surface free energy. Formation of additional features is energetically unfavorable since it increases the total fracture surface area. Therefore, except in the grain boundary affected zone, the fracture surfaces can be quite smooth [8].

Figure 2 shows that, when a river marking is relatively deep, i.e. as the height difference of the two cleavage planes across it is large, it can keep developing across a grain boundary, except that the direction varies, normal to the crack front propagating in the grain ahead of the boundary. The cleavage front segments at both sides of the cleavage ridge penetrate through the boundary in a similar way as that shown in Fig. 1.

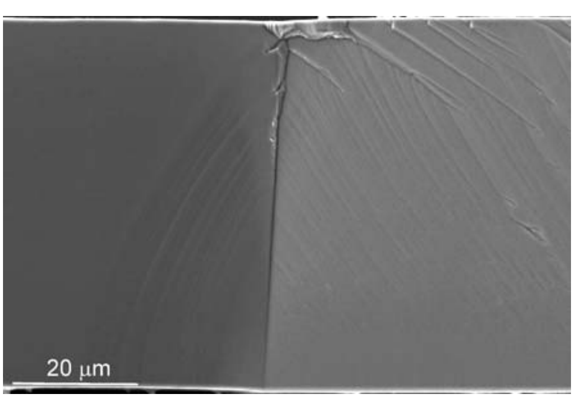

FIGURE 1 Environmental electron scanning microscopy of a cleavage crack of smooth surface

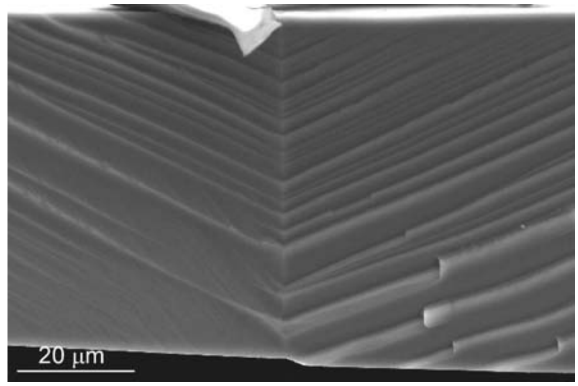

FIGURE 2 Microscopy of a cleavage crack of sectioned surfaces. The cleavage ridges bypass the grain boundary directly. The crack propagates from the *right* to the *left*

That is, the central part of the front section first enters the next grain, and then the sections next to the river marking follow, after the grain boundary is separated apart. The boundary separation does not affect the continuity of the secondary cracking plane. Under this condition, the numbers of river markings in the two grains across the boundary are nearly identical. If the depth of river markings reaches the saturation level, the change in roughness of fracture surface associated with crack front transmission would be negligible.

Clearly, since the river markings are deep, the cleavage facets separated by them behave quite independently. The initiation of grain boundary failure starts at the break-through points that are relatively far away from the ridges, and thus the cleavage ridges may not have a pronounced effect on the condition of onset of front transmission. However, as river markings become deeper and closer to each other, the areas of secondary fracture surfaces in the interior of a grain and at a boundary would increase, both of which demand larger work of separation. Thus, the river markings should be of a beneficial effect in increasing fracture toughness, primarily because of the increase in fracture surface roughness.

If the river markings are relatively shallow and their distance is small, the front segments interact with each other. As shown in Fig. 3a, their behaviors are quite similar with that shown in Fig. 2. Both of the grains are highly sectioned. The river marking distances are nearly the same, while their directions are different. The sudden change in river marking direction takes place at the grain boundary. Inside each grain, the river markings are quite straight, indicating that the cleavage front is nearly straight as it propagates. Approximately, there is a one-to-one match of cleavage ridges in the grain ahead of the boundary (grain "B") and the grain behind the boundary (grain "A"). However, they are not continuous. With respect to the corresponding cleavage ridge in grain "A", each ridge in grain "B" shifts by a small distance along the boundary, as if a kink were formed when the ridge extends across the boundary. Figure 3b shows a clearer image of such a cleavage ridge. In grain "A", there are a few parallel cleavage facets that are separated by river markings. When the river markings reach the boundary, they stop. New river markings in grain "B" are initiated at the boundary, and they are not directly connected with those in grain "A". Accordingly, the cleavage facets are also discontinuous at the boundary. The cleavage

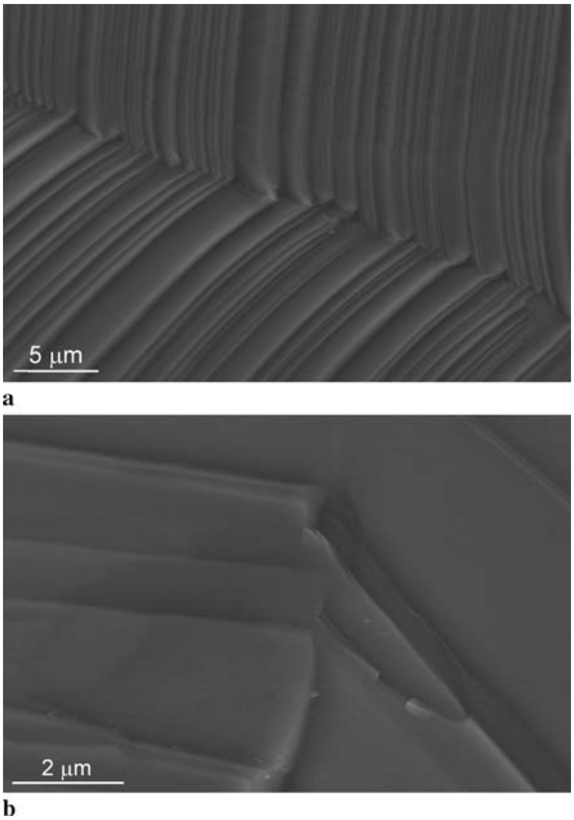

FIGURE 3 **a** Microscopy of a cleavage crack of sectioned surfaces. The cleavage ridges promote crack front transmission. The crack propagates from the *top* to the *bottom*. **b** Microscopy of a cleavage ridge at a grain boundary. The crack propagates from the *left* to the *right*

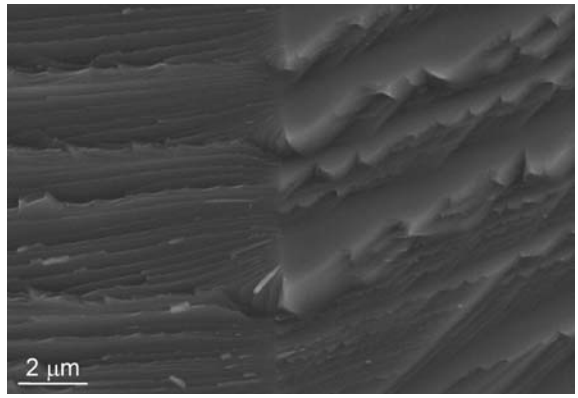

FIGURE 4 Microscopy of the smoothening process of a fracture surface. The crack propagates from the *left* to the *right*

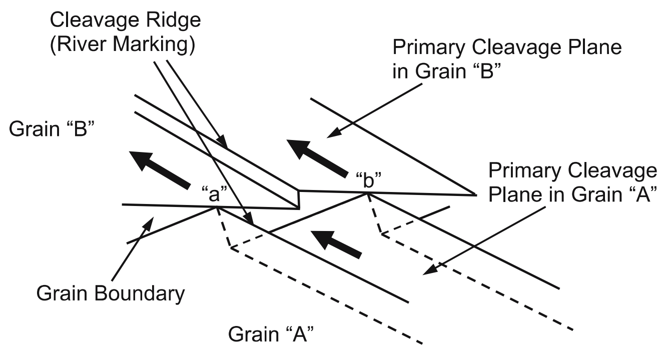

FIGURE 5 A schematic diagram of the penetration process of a sectioned cleavage front at a grain boundary

front segment between two river markings does not directly transmit into grain “B”. It stops at the boundary, and the new cleavage planes in grain “B” are initiated at the intersections of the boundary and the river markings in grain “A”. That is, the river markings promote cleavage facet nucleation, before the existing crack front can penetrate into the next grain.

This process is depicted in Fig. 5. The crack propagates from the right to the left. In grain “A”, the front branches into a few segments. The secondary crack surfaces connecting them are river markings. When the crack reaches the boundary, due to the barrier effect, it cannot enter grain “B” until the crack growth driving force further increases. According to both theoretical analysis [22] and computer simulation [23], at a ridge in the crack front the local stress intensity factor is much higher. Therefore, before the cleavage front segments in between them can trigger fracture in grain “B”, cleavage facets are nucleated at points “a” and “b”. This is different from the continuous extension of river markings. In this case, the cleavage ridges in grain “B” are formed when the fracture facets starting from “a” and “b” meet. Although there is still a one-to-one match, the heights of the river markings across the boundary can be quite different. That is, while the fracture appearance may not vary much, the effective roughness can change. The roughness may either increase or decrease, depending on the crystallographic orientations. Under either condition, the resistance offered by the boundary to a sectioned crack should be lower than that of a smooth crack.

Another interesting phenomenon observed in the experiment is that in some samples the river markings can be “smoothened” when the crack front bypasses a grain boundary (Fig. 4). In such a sample, the cleavage ridges do not directly trigger crack nucleation in grain “B”. The behavior of front segment in between ridges is similar with that shown in Fig. 2. That is, the segments act somewhat independently. They penetrate across the boundary at different heights. However, the cleavage ridges do not extend into the next grains. The front segments bow into grain “B”, becoming curved at borders. As a result, series of relatively small ridges are produced, “smoothly” connecting the new segments across the boundary. It can be seen that associated with the break-down of large river markings, the roughness of the fracture surface of grain “B” is much smaller. In these samples, the boundary toughness should be relatively small because less fracture work is required to separate the fracture flanks.

4 Conclusions

In summary, through a microtensile experiment on polysilicon thin films, three interaction modes of cleavage ridges with grain boundaries are identified. When the cleavage ridges in the grain behind the boundary are relatively deep and far apart from each other, the front segments between them transmit across the boundary quite independently. If the river markings are small and close to each other, they can trigger cleavage facet nucleation in the next grain. While the numbers of river markings are about the same, the ridge depth in the two grains can be quite different. Due to the promotion effect of sharp corners, the barrier effect of grain boundaries can be reduced. Finally, if secondary separation can take place along the boundary direction, the river markings can be smoothened.

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