HVEM/AFM Observation of Hinge-Type Plastic Zones Associated with Cracks in Silicon Crystals

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Dislocation structures developed in hinge-type plastic zones associated with cracks in silicon crystals have been studied using a high voltage electron microscope (HVEM). Fine slip bands due to those dislocations have been also examined by an atomic force microscope (AFM).

Cracks were introduced into (110) silicon wafers at room temperature by Vickers indentation method. The temperature of the wafer chips indented was raised to higher than 823 K to activate dislocations around crack tips under a residual stress due to the indentation. In specimens with the heat-treatment, prominent dislocation arrays corresponding to the hinge-type plastic zone were observed not only near the crack tip but also in the crack wake. AFM observations showed that very fine slip bands with the step height of a few nano-meters were formed with the regular spacing of a few microns. Based on the analyses of those dislocations and slip bands, it has been revealed that those dislocations were shielding-type increasing the fracture toughness.

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1. Introduction

It is well known that the brittle-to-ductile transition (BDT) of materials is controlled by dislocation behavior around the tip of a crack. Silicon crystals have been employed as a model substance to understand such dislocation process, since highly perfect crystals are obtainable and they exhibit a very sharp BDT.1, 2) When the BDT occurs to cause a steep increase of fracture toughness, a burst of dislocation multiplication takes place around the crack tip. However, TEM studies for such dislocation process in Si have been very limited so that the mechanism of the sharp BDT has been still unclear from the viewpoint of the dislocation theory.

Plastic zones associated with cracks under a mode I tensile load are macroscopically separated into two groups:3) the 45°-shear-type and the hinge-type (see Fig. 1). The former is well developed under the plane stress condition, the later under the plane strain condition. In the previous work, we reported dislocation configurations and fine slip bands associated with the 45°-shear-type plastic zone in [100] Si wafers.4, 5) In the present study, with the aim at examining the micro-structural characteristics of the hinge-type plastic zone, we have made both HVEM and AFM observations around a [100] crack using [110] silicon wafers where slip bands corresponding to the hinge-type plastic zone are well formed.

2. Experimental

We employed Si single crystals cut out from a [011] p-type wafer. Cracks were introduced at room temperature by Vickers indentation on a (011) surface. Two kinds of cracks were introduced from the edges of the indent: one is the [100] crack and the other is [110], as shown in Fig. 2. The specimen temperatures were raised to those between 823 K and 923 K to activate dislocations under a residual stress due to the indentation. After the heat-treatment, specimens were chemically polished and a selected area including a crack tip was thinned by focused ion beam (FIB), which was originally developed by Saka and Nagaya for plan-view observations of cracks in Si crystals.6) Observations of slip bands were carried out using an atomic force microscope (SII, SPI-400). TEM observations of cracks and dislocations were made using a high voltage electron microscope (JEOL, JEM-1000) at the Research Laboratory of High Voltage Electron Microscopy, Kyushu University.

3. Results and Discussion

Figures 3(a) and (b) show the AFM images of surface steps around a crack tip in a specimen before and after the heat treatment at 923 K, respectively. The plane observed is the (011) wafer surface. In both figures, the same (100) crack

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Fig. 2 Optical micrograph of an indent on the (011) surface. (100) and (01\bar{1}) cracks are introduced from the edges of the indent.

Fig. 3 AFM images around a crack introduced by indentation method in a (011) wafer. (a) without any heat treatments, (b) after annealing at 823 K for a few minutes, (c) schematic illustration of the crack and slip observed by AFM, (d) cross sectional profile along the arrow in Fig. (b).

is seen along the [01\bar{1}] direction. In Fig. 3(a), however, no steps are observed except those due to the crack and some contamination, indicating that the crack tip plasticity in silicon is negligible at room temperature. This was confirmed also by HVEM observation in the previous work. On the other hand, in Fig. 3(b), slip bands are formed with regular spacing of a few microns in the both sides of the crack. The traces of the slip bands mainly observed coincide with those of the (1\bar{1}1) or (1\bar{1}1) planes on the observed (011) surface, indicating that the slip bands correspond to those formed in the hinge-type plastic zone as illustrated in Fig. 3(c). A cross sectional profile of the surface step due to one of the slip bands is shown in Fig. 3(d), which was obtained by sectioning along an arrow indicated in Fig. 3(b). The left end of the abscissa in Fig. 3(d) corresponds to the location of the start point of the arrow in Fig. 3(b). In Fig. 3(d), a step with the height of 2.7 nm is seen, which is considered to be formed by the motion of about 14 dislocations since the height of a surface step formed by one dislocation is 0.19 nm. It is to be noted in Fig. 3(d) that the level of the surface in the left side of the slip band is higher than that in the right side. The same characteristics were seen in all the other slip bands in the left side of the crack. In the right side of the crack, the characteristic of slip bands appears symmetrical to that in the left side with respect to the crack plane, as shown in the illustration in Fig. 3(c). In addition, note in Fig. 3(b) that fine bands of other slip systems are also seen along the direction of the crack extension although the contrasts are not so clear as those due to the hinge-type. The trace of those slip bands is parallel to that of (1\bar{1}\bar{1}) or (1\bar{1}1) plane, indicating that they correspond to those due to \(45^\circ\)-shear-type plastic zones as shown in Fig. 1(b). Thus, the main component of the plastic zone observed in Fig. 3(b) is the hinge-type, although the \(45^\circ\)-shear-type is also included as a minor component.

In order to examine dislocation structures developed in the hinge-type plastic zone, HVEM observations were carried out. Figures 4(a) and (b) show HVEM images of dislocation arrays in the hinge-type plastic zone formed in a specimen subjected to the heat treatment at 823 K. In Fig. 4(a), the incident beam direction is the same as that of the foil normal, i.e. [01\bar{1}], while in Fig. 4(b) it was taken to be [00\bar{1}] by tilting the specimen in the holder. The diffraction vectors \(g\) in Figs. 4(a) and (b) were taken to be [02\bar{4}] and [\bar{4}00], respectively. Thompson tetrahedrons are illustrated in the right of each figure. A crack is extending along the [01\bar{1}] direction. Under the condition of the [01\bar{1}] incidence (Fig. 4(a)), the crack is observed as a line image, indicating that the crack plane is (100). In both Figs. 4(a) and (b), two prominent arrays of dislocations are seen horizontally: they are formed along the (1\bar{1}1) plane with the spacing of about three microns. As is seen in Fig. 4(b), the line segment of each dislocation in the arrays tends to lie along the [01\bar{1}] direction, although it is curved and seems to be bowing out from the right to the left. This suggests that the dislocation arrays are activated from sources on the crack by increasing temperature under the presence of residual stresses due to the indent. In order to confirm this consideration, we made characterization of these dislocations to determine the directions and the signs of their Burgers vectors. The characterization was made not only by the usual invisibility criterion but also by contrast simulations of dislocation images under various diffraction conditions. Although the details will be published in a separate paper, it was found that all dislocations in the arrays had the Burgers vector of \(a/2[\bar{1}0\bar{1}]\). Since their dislocation lines tend to lie along [01\bar{1}] as indicated above, they are mainly \(60^\circ\) dislocations. The sign of their Burgers vectors is shown in Fig. 5: extra-half planes of edge components are indicated in the figure.
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Fig. 4 HVEM images of dislocation arrays in the hinge-type plastic zone. Bright field images. Foil normal is [011]. (a) [011] incidence, $g = 02\bar{2}$, (b) [001] incidence, $g = 400$.

Fig. 5 Schematic illustration of the dislocation configuration observed in the slip bands of hinge type. A Thompson tetrahedron is also shown.

Fig. 6 (a) Dislocation configuration employed for the calculation of crack-dislocation interaction, (b) Contour map of a stress field (stress component normal to the crack plane) induced by dislocation arrays shown in Fig. (a).

ure, and the screw components are right-handed (R-H). If dislocations of this type glide away from the crack tip, crack tip deformation occurs as shown in Fig. 3(c).

In order to evaluate the effect of this type of dislocations on the fracture toughness, we calculated a local stress intensity factor due to those dislocations. In this calculation, we made the following simplification: 1) dislocation lines are parallel to [011], i.e. the foil normal. By this simplification, the crack tip and the dislocation lines are parallel to each other, so that the crack-dislocation system is treated as a two dimensional problem, 2) dislocations are emitted symmetrically with respect to the crack plane. As is indicated in Fig. 5, the dislocations observed in the hinge-type plastic zone in the present study are 60° dislocations. This indicates that the edge component of those dislocations is 40% larger than that of screw, i.e., the main component is the edge. In addition, when the dislocation lines are parallel to the tip of a crack, the screw component of dislocations emitted from a crack tip contributes only to a mode III stress intensity, which is not a main field causing usual materials fractures. In the present work, therefore, only the edge component was taken into account for the calculation of crack-dislocation interaction. A crack-dislocation model employed is illustrated in Fig. 6(a). Here, dislocation lines are parallel to [011] and they are lying on the (111) or the (111) slip planes. Burgers vectors of the dislocations are assumed to be $(\sqrt{3}/4)a[110]$ since only the edge component is considered. Thirty dislocations are placed on each slip plane and the distance between adjacent dislocations is put to be 0.33 μm, which is based on the results of
the present observations. The contour map of crack-tip stress field calculated is shown in Fig. 6(b), where the component of the stress field is that normal to the crack plane: \(\sigma_{yy}\) in the coordinate system in Fig. 6(a). In the calculation, no external stress was applied, so that all the stresses seen in the figure are due to the internal stresses of dislocations around the crack. The shaded region in Fig. 6(b) indicates negative (compressive) stresses. Note in this figure that a stress concentration appears at the crack tip in spite that no external stress is applied. Moreover, the stress concentration at the crack tip is compressive, which suggests that the stress concentration can be relaxed (shielded) by the compressive stress when external tensile stress is applied. The magnitude of the local stress intensity factor (mode I) due to the dislocations, \(k_{ID}\), was calculated to be \(-0.722\) MPa·m\(^{1/2}\). The negative value of \(k_{ID}\) contributes to the increase of fracture toughness, although the magnitude of \(k_{ID}\) is rather smaller than the increment of fracture toughness at BDT in Si crystals.\(^1\) Therefore, dislocation structures observed in the present study is considered to correspond to that in the early stage of the BDT.

4. Conclusion

Crack tip dislocations and slip bands formed in the hinge-type plastic zone in Si crystals were observed by HVEM and AFM. No dislocations were observed in a specimen where a crack extension occurred at room temperature. When the temperature of a (011) wafer chip including (100) cracks was raised to more than 823 K, plastic deformation occurred around the crack to form hinge-type plastic zone. By using AFM, very fine slip bands with the step height of about 3 nm were observed around the crack. Those slip bands were formed not only from the crack tip but also in the crack wake with the regular spacing of a few microns. In the slip bands, sharp arrays of 60\(^\circ\) dislocations were observed. The dislocations contribute to the increase of fracture toughness, although the increment of fracture toughness is smaller than that at BDT. This suggests that the observed dislocation structure corresponds to the early stage of BDT.

REFERENCES