Article

Crack tip shielding observed with high-resolution transmission electron microscopy

Damar Rastri Adhika¹, Masaki Tanaka¹.*, Takeshi Daio², and Kenji Higashida¹

¹Department of Materials Science and Engineering, Kyushu University, 744 Motooka, Nishi-ku, Fukuoka 819-0395, Japan, and ²Department of Hydrogen Energy Systems, Kyushu University, 744 Motooka, Nishi-ku, Fukuoka 819-0395, Japan

*To whom correspondence should be addressed. E-mail: masaki@zaiko.kyushu-u.ac.jp

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Abstract

The dislocation shielding field at a crack tip was experimentally proven at the atomic scale by measuring the local strain in front of the crack tip using high-resolution transmission electron microscopy (HRTEM) and geometric phase analysis (GPA). Single crystalline (110) silicon wafers were employed. Cracks were introduced using a Vickers indenter at room temperature. The crack tip region was observed using HRTEM followed by strain measurements using GPA. The measured strain field at the crack tip was compressive owing to dislocation shielding, which is in good agreement with the strain field calculated from elastic theory.

Key words: HRTEM, brittle-to-ductile transition, dislocation shielding effect, fracture toughness, geometric phase analysis

Introduction

When stress is applied to a crack introduced in ductile materials, dislocations are activated and propagate from the crack tip. The stress concentration at the crack tip is accommodated by dislocation activities such as dislocation glide and multiplication, which lead to an increase in fracture toughness. Fracture toughness increases with temperature, and the fracture mode changes from brittle to ductile, which is the so-called brittle-to-ductile transition (BDT). Silicon single crystals have been used as model crystals to understand the mechanism behind the BDT because they show an abrupt increase in the fracture toughness at a certain temperature and because large dislocation-free silicon single crystals are available. St. John [1] pointed out that the increase in the fracture toughness at the BDT is controlled by dislocation glide in silicon. Hirsch et al. [2] suggested that the abrupt increase in the fracture toughness at the BDT in silicon could be explained by the assumption that the value of the critical stress intensity factor for dislocation emission at the crack tip should be close to that of the fracture toughness of silicon. A gradual increase in fracture toughness, in contrast, could be due to the low critical stress intensity factor for dislocation emission. These findings indicate that there is a direct relationship between dislocations and the BDT. Figure 1(a) shows a contour map of the stress component normal to the crack plane, $\sigma_{yy}$, around a crack tip, with a pair of pure edge dislocations in front of the crack plane, $\sigma_{yy}$, around a crack tip, with a pair of pure edge dislocations in front of the crack tip without any stress applied [3,4]. The contour map was obtained by calculating the stress field based on the elastic theory derived by Thomson, with the dislocation configuration as shown in Fig. 1(b). In this configuration, two dislocations are located $\pm 45^\circ$ from the crack line and...
100 μm away from the crack tip [4]. The extra half-planes of these dislocations were both at the crack side with respect to their slip planes, as shown in Fig. 1(b), which represents the situation after two pure edge dislocations have been emitted from the crack tip. The shaded area around the crack tip in Fig. 1(a) represents a compressive stress field. The emitted dislocations shield the crack tip. This indicates that some extra tensile stress is necessary for the crack to propagate, cancelling the compressive stress concentration due to the dislocations. This is the origin of the increase in the fracture toughness from the viewpoint of crack tip shielding due to dislocations [2,5–8].

The first macroscopic experimental verification of the shielding effect due to dislocations was reported using photoelasticity in ionic crystals [4]. It was shown that the residual stress field around a crack after deformation was actually a compressive stress field. In addition, Higashida et al. directly observed crack tip dislocations in MgO and silicon single crystals after deformation. The Burgers vectors, including their signs, were determined in this study using high-voltage electron microscopy [9–12]. They pointed out that the local stress intensity factor due to dislocations calculated from the dislocation configuration is negative, which indicated that the dislocations observed were of the shielding type.

A recent development in high-resolution transmission electron microscopy (HRTEM) makes it possible to measure strain at the atomic scale. Hýtch [13] suggested geometric phase analysis (GPA) which enables the calculation of the strain distribution from HRTEM images. GPA utilizes the crystal periodicity information that is extracted by performing Fourier transformations of HRTEM images [13]. It is possible to measure the elastic displacement of atoms from a non-deformed area by calculating the change in phase of the lattice image. Once the displacement of atoms is known, the elastic strain field due to the displacement of atoms can be calculated. In this study, the local strain in front of a crack tip with dislocations in single crystal silicon was measured using GPA software (HREM Research), which is a plug-in for DigitalMicrograph (Gatan), to clarify the existence of the shielding effect of dislocations at the atomic scale.

**Experimental method**

The sample used in this study was a (110) single crystal silicon wafer with a thickness of 0.63 mm. An indentation technique was employed to introduce cracks in the sample. Figure 2 shows an indent on the sample surface introduced using a micro Vickers indenter (MVK-E3, Mitutoyo) with an applied load of 0.5 N and a dwell time of 7 s. Cracks propagated from the corners of the indent along both the <100> and <110> directions. The {100} cracks that propagated along the <110> directions were shorter than the {110} cracks that propagated along the <100> directions. The difference in crack length indicates the difference in fracture toughness [14]. That is, fracture toughness on the {100} planes was higher than that on the {110} planes [15]. Because {110} is the second cleavage plane of silicon, it is essential to elucidate the strain around {110} crack tips with dislocations. Therefore, one of the {110} crack tips was observed in this study.

The TEM sample used in this study was prepared using focused ion beam (FIB, Quanta 3D 200i, FEI). First, carbon was deposited on the top surface to protect the crack from damage by ion beams during the machining process. The area around the deposited region was milled with an ion beam at an operating voltage of 30 kV. The TEM sample was then lifted out and attached to a mesh. The protective carbon layer was removed and the sample was thinned by an ion beam with an operating voltage of 30 kV, which was gradually lowered to 16, 8, 5 and 2 kV. The thinning at smaller voltages was performed to remove layers damaged during the higher voltage thinning process. The sample was...
thinned until the maximum thickness of the sample was <200 nm. Figure 3 shows a HRTEM sample after FIB machining, wherein a crack is extending from the left edge to the centre. The crack is located in a very small area that is about 480 nm away from the thinnest edge. The sample was then subjected to post-FIB cleaning using a NanoMill system (Model 1040, Fischione). This cleaning was conducted with an operating voltage of 900 V for 5 min at a tilting angle ±10° to clean the top and bottom of the sample. It was followed by milling with 500 V for 10 min on each side to further reduce damage. The final sample thickness around the crack tip was ∼65 nm, which was measured during TEM observation using the electron energy loss spectroscopy (EELS) log-ratio technique.

The sample thickness, \( t \), is given by the intensity of the zero-loss electron, \( I_o \), and the total electron intensity, \( I_t \), in an energy loss spectrum as follows [16]:

\[
t = \lambda \ln \left( \frac{I_t}{I_o} \right)
\]

and

\[
\lambda = \frac{106 \times F \times E_o}{E_m \ln \left( \frac{2BE_o}{E_m} \right)}
\]

where \( \lambda \) is the mean free path of electrons for inelastic scattering, \( F \) is the realistic correction factor, \( E_o \) is the incident energy of electrons, \( E_m \) is the average energy loss of electrons and \( \beta \) is the collection angle.

The foil sample was observed with an aberration corrected HRTEM (JEM-ARM200F, JEOL) at an operating voltage of 200 kV at the Ultramicroscopy Research Center, Kyushu University.

Results and discussion

Figure 4(a) shows a low-magnification TEM image indicating the position of a crack extending from the edge of the foil sample. To ascertain the exact position of the crack tip at the atomic scale, the crack tip was gradually magnified. It is also necessary to use high-angle annular dark field scanning transmission electron microscopy (HAADF-STEM) to determine the exact position of the crack tip. Figure 4(b) shows a Z-contrast HAADF-STEM image in which the dark area is the crack, which is clearly distinguishable from the matrix. Figure 4(c) shows an enlarged HAADF-STEM image of the area indicated by an arrow in Fig. 4(b) at the atomic scale, indicating that the crack is observed nearly...
edge-on. The crack plane is (011) and the crack is opened 2 nm wide.

Figure 5(a) shows an original HRTEM image of the crack tip at the atomic scale in the same area as that in Fig. 4(c). The black region of this image indicates the crack, as determined by Fig. 4(c). The distorted area in front of the crack tip indicates the existence of dislocations in that region. To determine the character of those dislocations around the crack tip, the original image was filtered to clarify lattice planes. First, the original HRTEM image was transformed into the frequency domain using the Fourier fast transformation (FFT), as shown in Fig. 5(c). The background, or low frequency noise, in the centre spot of the FFT image was filtered out. The information from higher frequencies was captured by applying masks. The largest mask size of 0.75 nm\(^{-1}\) is shown in Fig. 5(d). The filtered FFT image in Fig. 5(d) was then converted back to image space by performing the inverse FFT, resulting in the filtered HRTEM image shown in Fig. 5(b). From Fig. 5(b) it could be seen that there are four dislocations around the crack tip: D1, D2, D3 and D4. The Thompson’s tetrahedron is also shown in the figure. There are two dislocations in the distorted region in front of the crack tip and two other dislocations on the left-hand and right-hand sides of the crack. Red and green lines in the figure represent the extra half-planes of each dislocation. The dislocations denoted as D1 and D3 lie on the slip plane of (111) while those denoted as D2 and D4 lie on the slip plane of (111). The Burgers vectors of the dislocations were also determined by using the finish-to-start-right-hand (FS/RH) convention [17]. Each Burgers circuit coloured in yellow in front of the crack tip contains one dislocation. These Burgers circuits were then transferred to undistorted regions at the right-hand and left-hand sides above the crack tip, forming open circuits. Because the image is the projection on the (011) plane, the exact directions of their Burgers vectors cannot be determined perfectly. The Thompson’s tetrahedron indicates the possible Burgers vectors for each dislocation. Dislocations D1 and D3 have the same possible pair of Burgers vectors, and so do dislocations D2 and D4. The possible Burgers vectors and their partial components are listed in Table 1. The Burgers vectors of D1–D4 are defined to be \(b_1\) to \(b_4\). In each case, the edge component of the Burgers vectors are the same for D1, D3; and D2, D4; which are \(a/6[2\bar{1}1]\) and \(a/6[2\bar{1}1]\), respectively. Here, we focus on the edge
component of the Burgers vector, which induces mode I shielding at the crack tip. To clarify the dislocation shielding field at the atomic scale, the strain field around the crack tip was measured using GPA.

Figure 6(a) shows ε_{yy} around the crack tip depicted in Fig. 5. The blue areas indicate compressive strain fields while the red areas indicate tensile strain fields. There are four butterfly-like points around the crack tip where the blue and red colours discontinuously changed. The value of strain is divergent where the elastic theory is not applicable. The strain field around the crack tip is also divergent. It should be noted here that ε_{yy} is negative around the crack tip, that is, the stress intensity at the crack tip is compressive, which is caused by those dislocations in front of the crack tip.

To verify the experimental results, the crack tip strain field with the dislocations was also calculated using analytical equations derived by Rice and Thomson [18]. The strain components in two-dimensions at the crack tip with dislocations, but without external stress, are given by the following equations, where the crack plane lies along the x-axis and the crack tip is at the origin of the complex field [17,18]:

\[
\begin{align*}
\epsilon_{xx} &= \frac{1}{E} \sigma_{xx} - \nu (\sigma_{xy} + \sigma_{zz}), \\
\epsilon_{yy} &= \frac{1}{E} \sigma_{yy} - \nu (\sigma_{xx} + \sigma_{zz}), \\
\epsilon_{xy} &= \frac{1}{2\mu} \sigma_{xy}, \\
\sigma_{xx} + \sigma_{yy} &= 2 [q'(z) + q'(\bar{z})], \\
\sigma_{yy} - i \sigma_{xy} &= q' + \bar{q}' + (z - \bar{z})q'' \\
\end{align*}
\]

where z is a complex variable for the position relative to the crack tip in the form of \( z = x + iy \), \( \zeta_j \) is the position of the \( j \)th dislocation in the form of \( \zeta_j = Cx + iDy \), \( \mu \) is the shear modulus (64 GPa for silicon), \( b_j \) is the Burgers vector of the \( j \)th dislocation in the form of \( b_j = Fx + iGy \) and \( \nu \) is the Poisson’s ratio (0.278).

The strain field around the crack tip was calculated with four dislocations and without applied stress. The positions and the Burgers vectors are those of the experimentally
observed dislocations shown in Fig. 5. Figure 6(b) shows the calculated strain map of $\varepsilon_{yy}$ in the same spatial scale as that in Fig. 6(a). Figure 6(b) also shows compressive strain fields at the crack tip and at the dislocation cores. The calculated strain map is in good agreement with the experimental result shown in Fig. 6(a). It is stressed that these results show experimental proof of the dislocation shielding effect observed at the atomic scale in front of the crack tip.

Concluding remarks

The crack-tip strain field with dislocations was measured using HRTEM and GPA. The measured strain field around the crack tip was compressive owing to the dislocation shielding effect. The measured strain field was in good agreement with that calculated from elastic theory. These results show experimental proof of the dislocation shielding effect at the atomic scale. The dislocations create an extra barrier in front of the crack tip, making it more difficult for the crack to propagate, which increase the fracture toughness at the BDT.

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