Tensile Deformation of Si Single Crystals with Easy Glide Orientation

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1. Introduction

Stress–strain (s–s) curves from single crystals comprehensively express the process of work-hardening in crystals. The work-hardening process can be divided into three stages: stage I, II, and III. Generally, it is known that stages I and II show the lowest and highest work-hardening rates, respectively, while stage III shows a decrease in the rate of increase in the work-hardening rate. Many studies have investigated activated slip systems and dislocation structures in each stage, observing slip traces and dislocations using optical and electron microscopy.¹⁻³ It has been found that the length of the steps formed on a specimen surface in stage I can reach 600 µm or more. The long steps are formed because dislocations can glide long distance on the primary slip system without being disturbed. In stage II, the length of steps becomes shorter of 15 to 50 µm along the primary slip axis. The secondary slip system is also activated. Many dislocations form loops and networks. This suggests that the dislocations in the secondary slip system hinder the motion of dislocations in the primary slip system, decreasing the mean free path of the dislocation motion.⁴ Short slip traces along cross slip plane are also formed in stage III because of the increase in the possibility of cross slipp.⁴

As in this observation of slip bands, the increase in the density of dislocations with the plastic strain shortens the mean free path of the dislocation motion. Such dislocations in secondary slip systems should accumulate to some extent not only in stage II but also in stage I. They influence the motion of dislocations in the primary slip system to increase the deformation stress. The work-hardening processes and the existence of secondary slip system dislocations are closely related. Here, the deformation shear stress is given by the following relation:⁵

\[ \tau_d = \tau_0 + \alpha \mu b \rho^{1/2}, \]  

where \( \alpha \) is a constant (approximately 0.5), \( \mu \) is the shear modulus, \( b \) is the Burgers vector, \( \tau_0 \) is the friction stress, and \( \rho \) is the density of forest dislocations in the secondary slip system.

The effect of dislocations in the secondary slip system on work-hardening strongly depends on the orientation of the tensile axis, especially in stages I and II. It is known that the work-hardening rates of specimens in which the initial tensile axis is set to be near the zone axis, such as (100) and (111), are larger than those set to be near the center of the standard triangle such as (134).⁶ This is because dislocations on the secondary slip system can be activated even in the early stage of plastic deformation in the former case.⁷ Although the involvement of dislocations on the secondary slip system in work-hardening is clear, the transition from stage I to stage II, is still ambiguous.

The greater number of silicon is expected to be used as power devises in automobiles. The mechanical properties of silicon single crystals are now getting attention because silicon wafers sometimes deform during heat treatment during device processing, which reduces the productivity of devices. However, the number of studies on whole deformation behavior of Si is quite limited especially work-hardening while the yielding of silicon and the properties of dislocation motions have been well studied. Yonenaga et al. reported that temperature, initial dislocation density, and interstitial oxygen atoms have significant effects on the yield point.⁸⁻¹⁰ In addition to that, they found that not only the upper yield stress but also the lower yield stress are influenced by the initial dislocation density. Interstitial oxygen atoms do not significantly affect the work-hardening process after yielding, but it does significantly affect the yielding due to the locking of initial dislocations.¹¹⁻¹² The relationship between precipitated oxygen and yield behavior was also investigate. It was found that dislocations punched out from precipitates decreases the yield stress.

In the present study the transition from stage I to stage II in a single crystal was investigated employing single
crystalline silicon crystals that have the same slip system as fcc crystals. The rotation of the tensile axis during plastic deformation in single crystals was investigated by using electron backscattered diffraction (EBSD). The relationship between the rotation of the tensile axis at the onset of stage II and the activation of secondary slip systems was investigated in order to understand what triggers the onset of stage II in single crystals.

2. Experimental Methods

The tensile specimens used in this study were cut out from (111) Czochralski silicon wafers commercially available, which contain interstitial oxygen atoms of $12 \times 10^{17}$ atom/cm. The plane normal of wafers is slightly tilted from the exact (111) direction, so-called off-angle. Figure 1 shows the schematic of a tensile specimen. The initial tensile axis was chosen to be $\{134\}$ where a single slip occurred. Tensile tests were conducted in a vacuum chamber with an oxygen partial pressure of approximately $10^{-6}$ Pa at an initial strain rate of $4.7 \times 10^{-4}$ s$^{-1}$ at temperatures between 1173 K and 1373 K. The tensile tests were recorded with a CCD camera installed in the tensile machine (Yonekura CATY-T3H). Strain was measured between two points, the length of which is 2.0 mm, on specimen surfaces by using a software (Ditect, Dip-Motion). The change in the orientation was measured by the EBSD method in order to track the crystal rotation due to plastic deformation. Some tensile tests were interrupted at each stage of work-hardening for each specimen.

3. Results

Figures 2(a) and (b) show full s–s curves and those up to a total nominal strain of 0.2, respectively. The yield point phenomenon was observed in the specimens deformed below 1273 K, while it was not observed in the specimens deformed above 1323 K. This indicates that the dislocation density abruptly increases at yielding below 1274 K, while it gradually increases above 1323 K. The values of the tensile strength and the total strain at rupture are shown in Table 1, which indicates that the tensile strength decreased with increasing temperature.

The s–s curves show three definite stages in work-hardening at any test temperature, where stages I, II, and III show a low work-hardening rate after yielding, the highest work-hardening rate, and a decrease in the increase in the work-hardening rate, respectively. The work-hardening rates were measured by drawing a tangent line at the part where the work-hardening rate was constant in each stage not to be affected by the yield point and the transition of each stage as shown in Fig. 3. The transition strain of each stage is defined as the strain at the intersection of those tangent lines. Values of the transition strain are shown in Table 2 with the ranges

![Fig. 1 Schematic of a specimen with specimen coordinates of A1, A2, and A3.](image1)

![Fig. 2 (a) Stress–strain curves from the specimens tested at several temperatures. (b) Enlarged image of (a).](image2)

![Fig. 3 Means for measuring each value.](image3)

<table>
<thead>
<tr>
<th>Temperature (K)</th>
<th>Tensile strength (MPa)</th>
<th>Total strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>1173</td>
<td>144</td>
<td>0.57</td>
</tr>
<tr>
<td>1223</td>
<td>109</td>
<td>0.84</td>
</tr>
<tr>
<td>1273</td>
<td>86</td>
<td>1.67</td>
</tr>
<tr>
<td>1323</td>
<td>104</td>
<td>2.23</td>
</tr>
<tr>
<td>1373</td>
<td>92</td>
<td>1.91</td>
</tr>
</tbody>
</table>
of the strain measured to obtain work-hardening rate in each stage. The values of work-hardening rates of each stage and the total strain at the onset of stage II, and stage III are shown in Table 2. Colored arrows in Fig. 2(b) indicate the points at which stage II begins. This indicates that stage II begins at a lower strain as the test temperature is increased. Representing the shear modulus of silicon as \( \mu \) (approximately 80 GPa), the work-hardening rate in stage II is approximately \( 4 \times 10^{-3} \mu \approx 7 \times 10^{-3} \mu \), which corresponds well to the values reported for other single crystals so far.\(^{14,15}\) The work-hardening rate in stage III was approximately \( 6 \times 10^{-4} \mu \approx 1 \times 10^{-3} \mu \), which is lower than the rates obtained in stage II. The values in stage III also correspond well to those obtained for other single crystals reported so far.\(^{14,16}\) It is known that the values of work-hardening rate of fcc and bcc crystal are close.\(^{17}\) The fact that the work-hardening rates are identical whereas the crystal structure indicates that the mechanism behind the work-hardening is also identical. The work-hardening rates are not very sensitive to temperature in stages II and III.

<table>
<thead>
<tr>
<th>Temperature (K)</th>
<th>Work-hardening rate [Range of the strain measured] (MPa)</th>
<th>Total strain at the onset of each stage</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Stage I</td>
<td>Stage II</td>
</tr>
<tr>
<td>1173</td>
<td>163 [0.03-0.12]</td>
<td>544 [0.19-0.27]</td>
</tr>
<tr>
<td>1223</td>
<td>99 [0.03-0.12]</td>
<td>546 [0.14-0.17]</td>
</tr>
<tr>
<td>1273</td>
<td>100 [0.02-0.07]</td>
<td>522 [0.09-0.14]</td>
</tr>
<tr>
<td>1323</td>
<td>103 [0.01-0.03]</td>
<td>583 [0.06-0.09]</td>
</tr>
<tr>
<td>1373</td>
<td>174 [0.01-0.03]</td>
<td>554 [0.07-0.08]</td>
</tr>
</tbody>
</table>

Table 2 Work-hardening rate and total strain for the onset of stages II and III.

Figure 4 shows orientation maps and inverse pole figures with respect to the A3 and A2 directions from the gauge section after fracture tested at 1223 K in the specimen after fracture. The initial orientations of A1, A2, and A3 were [752], [134], and [111], respectively. Figure 4(a) indicates that the sample was ruptured at the right end of the figure after necking. The A3 direction was maintained to be parallel to the [111] direction, which was the initial orientation, until the specimen fractured, while the direction of the tensile axis (A2) rotated toward the [112] direction during the tensile test, as shown in Fig. 4(b). Figures 4(c) shows an inverse pole figure centering [001] direction of the specimen after the tensile test, and Figs. 4(d) and (e) show inverse pole figures with respect to the A2 direction of the specimen before and after the tensile test, respectively. The red curve in (e) indicates a great circle of the [111] zone axis, showing that the tensile axis direction (A2) is dispersed from the initial tensile direction during deformation. The dispersed points in Fig. 4(e) are on the [111] zone axis, which indicates that the rotation axis is the [111] direction. It also indicates that the
whole specimen was not uniformly rotated, i.e., the crystal direction pointing to the tensile axis (A2) is different from place to place inside the specimen. The tensile axis was dispersed along the great circle connecting [134] and [112] in the standard triangle. It also overshoots from the standard triangle to the next triangle surrounded by [101], [011], and [111]. Because there are many plots near the [112] direction, it is expected that the reverse rotation of the tensile axis toward the [011] direction due to the activation of the conjugate slip after the tensile axis penetrates into the triangle of [101]-[101]-[001]. It has been considered that stage II begins with the onset of multiple slips.\(^{18-20}\) Therefore, it is expected that stage II begins when the Schmid factor of the conjugate slip becomes higher than that of the primary slip when the tensile direction overshoots the great circle connecting [001] and [111].

In order to elucidate the relationship between the onset of stage II and the activity of the conjugate slip system, tensile tests at 1273 K were terminated at two different value of strain during the deformation: at the end of stage I and stage II shown in Fig. 5(a). The change in the tensile direction inside the specimen was investigated by EBSD. Figures 5(b) and (c) show orientation map with respect to the A2 direction, which were obtained after unloading at the end of stage I (total strain of 0.09), and at the end of stage II (total strain of 0.29), respectively. Note that the data in Figs. 5(b) and (c) were taken from exactly the same area of the specimen. Figure 5(d) shows an optical micrograph of the specimen shown in Fig. 5(b), indicating slip traces on the specimen. The angle between the traces (shown with red dashed lines) of the slip traces and the tensile axis direction (A2) was 34°. This value is nearly the same as 35°, which is the trace angle of the primary slip plane of (111) to the plane on the specimen surface of (111). Therefore, also determining from the magnitudes of Schmid factor, it is clear that the primary slip system (111)[101] was activated in stage I. A schematic of Thompson’s tetrahedron is also shown in Fig. 5(b). The slip plane is colored in red and the slip direction is shown with a green arrow on the tetrahedron. Note, that the slip traces of the slip direction [101] for the primary slip cannot be observed from the (111) surface because the Burgers vector is on the (111) plane. The reason why the slip traces were observable in the specimen surface is that the {111} Si wafer used in this study was one

![Fig. 5](image-url)
commercially available. It is known that the plane normal of wafers is slightly tilted from the exact (111) direction, so-called off-angle, to some advantages in the device processing.

Figure 5(e) shows an optical micrograph of the specimen shown in Fig. 5(c), indicating two types of slip traces on the specimen surface. The remarkable slip traces indicated by red dashed lines are the same as those seen in Fig. 5(d), which developed during additional deformation applied for the specimen shown in Fig. 5(c). Another type of the slip band is also seen in Fig. 5(e). Figure 6 shows an enlarged image of the slip bands observed with backscattered electrons. The angle between the slip traces parallel to the blue dashed line in Fig. 6 and the tensile axis direction (A2) was approximately 36°. Schematics of Thompson’s tetrahedron are shown in Fig. 5(c). The secondary slip plane is colored in blue and the slip direction is indicated by a yellow arrow in the tetrahedron. The tetrahedron indicates that the expected angle between the slip traces of the conjugate (secondary) slip system and A2 was approximately 30°, which is in good agreement with the angle of 36° shown in Fig. 6 although there is slight difference in the angle due to the rotation of the specimen. This indicates that the introduced slip traces in Fig. 6 were formed by the activation of the conjugate (secondary) slip system of (111)[011].

Figure 5(f) shows an inverse pole figure with respect to the tensile axis direction (A2), which was obtained from the region surrounded by a dashed white rectangle in Fig. 5(b). In the inverse pole figures in Figs. 5(f) and (g), × denotes an initial crystallographic orientation parallel to the A2 direction. This indicates that the tensile axis direction is still within the standard triangle even at the end of stage I, i.e., the beginning of stage II. It has been considered that the onset of stage II is significantly influenced by the activity of the conjugate slip system.21) The crystallographic orientation of the loading axis was determined from the inverse pole figure with respect to the tensile axis direction (A2) shown in Figs. 5(f) and (g). The A2 direction was set to be consistent during the tensile tests, so that black dots in Figs. 5(f) and (g) indicate the crystallographic orientation measured with 10 µm intervals inside the rectangles in (b) and (c). The average direction of these points was determined as the loading axis orientation in this test. The local misorientation inside the restables in (b) from the tensile direction at the end of stage II is no more than 3°. If the stress state of the tensile test is uniaxial along the tensile direction, it is expected that the tensile direction should have moved into the triangle of [101]-[011]-[111] where the Schmid factor of the conjugate slip system is higher than that of the primary slip. However, the conjugate slip system was already activated even though the tensile direction was still inside the standard triangle, as shown in Fig. 6, where the Schmid factor of the primary slip system is still higher than that of the conjugate slip system, as shown in Table 3. In other words, the conjugate slip system was activated, whereas the Schmid factor for the second slip system was not large enough. This indicates that the onset of the conjugate slip system cannot be understood only in terms of the Schmid factor.

4. Discussion

Assuming that only the primary slip system is active, the length of the specimen is given by the following relation:

$$l = \frac{(1 + \Delta l) \sin \theta}{\sin \theta_0},$$

where $l$ is the initial length of the specimen, $\Delta l$ is the increment of the length of the specimen, $\theta_0$ is the angle between the slip direction and the tensile axis, and $\theta$ is the angle between the slip direction and the tensile axis after deformation. Then, the angle between the tensile axis at the nominal strain of $\varepsilon$ and the initial tensile axis, $\lambda$, as follows:

$$\sin \theta_0 = \left(1 + \frac{\Delta l}{l} \right) \sin(\theta_0 - \lambda)$$

$$\lambda = \theta_0 - \arcsin \left(\frac{\sin \theta_0}{(1 + \varepsilon)}\right)$$

When the slip direction is [101] of the primary slip system and the initial tensile axis is [134], $\theta_0$ is 46°. Figure 7(a) shows the tensile direction when $\lambda = 0^\circ$ marked as × and $\lambda = 16^\circ$ marked as O. The tensile direction moves to the triangle of [001]-[111]-[101] when $\lambda > 16^\circ$.

Figure 7(b) shows the relationship between the Schmid factors of the primary slip system of (111)[101] and the conjugate slip system of [111][011] with respect to either $\lambda$ or $\varepsilon$. As $\lambda$ increases, the tensile axis rotates to the [101] direction with respect to the rotation axis of the [111]. The great circle with respect to the zone axis of [111] direction is also drawn as a red line in Fig. 7(a). Figure 7(b) indicates that the Schmid factor of the primary slip system is larger when $\lambda < 16^\circ$, i.e., $\varepsilon < 0.44$, while the Schmid factor of the conjugate slip system is large when $16^\circ < \lambda$, i.e., $0.44 < \varepsilon$. The values of $\lambda$ at the onset of stage II and the end of stage III (the onset of stage II) measured in the specimen in Figs. 5(f) and (g) were 9.5° and 11.6°, respectively. It is to be
noted that as also shown in Figs. 2, 5 and 6 that the conjugate (secondary) slip system was activated even when the tensile axis kept in the standard triangle where the Schmid factor of the primary slip system is the largest.

As mentioned above, the onset of stage II cannot be explained solely by the value of the Schmid factor. The change in the tensile direction due to the local rotation of the specimen during stage I and the subsequent stage II will be discussed with further analysis by EBSD about the specimen shown in Fig. 5(b), which was deformed to the end of stage I with a strain of 0.09. Figure 8(a) shows an optical micrograph with a slip traces along the red dashed line. Figure 8(b) shows a color map corresponding with a pole figure shown in (c). Blue and red plots in Fig. 8(c) were taken from blue and read areas in Fig. 8(b), respectively. It demonstrates that the distribution of plots in (c) is rotated with the common axis surrounded by a green circle, which is one of the (112) directions. Figure 8(d) shows a point-to-origin misorientation profile measured along a line indicated in (b), the origin of which is the point denoted as (I). Table 4 shows values of relative misorientations between two points. There are two boundaries showing relatively large misorientations near the grips while there are four boundaries showing the relatively low misorientations inside the outermost two boundaries with large misorientations. This indicates that these boundaries are so-called kink bands. A kink band is formed with a pair of planar arrays of edge dislocations, which are on the parallel primary slip plane and have the same Burgers vector as shown in Fig. 8(e). The dislocation line vectors of the edge dislocations are along

Fig. 7  (a) $\lambda$ on the inverse pole figure of the A2 direction. (b) Relationship between the Schmid factors and $\lambda$.

Fig. 8  (a) Optical micrographs of the gauge sections. (b) Color map using the plots in the inverse pole figure in (c). Arrows indicate kink boundaries. (c) Inverse pole figure with respect to A2. (d) Point-to-origin misorientation measured along a line in (b). (e) Schematic of a dislocation array at kink boundaries.
slip system of edge dislocations arrayed perpendicular to the activated deformation bands caused by non-uniform slip and are sets observed at the end of stage II. Kink bands are one of the Fig. 8(e). It should be noted here that clear kink bands were ½ rotation with respect to the single crystal and pointed out the formation of kink bands 25,26) The onset of stage II can be determined simply by the magnitude of the Schmid factor. (3) The direction of the tensile axis rotated toward [112] during the tensile tests. The crystallographic orientation of tensile axis was dispersed. This indicated that the crystal direction parallel to the tensile axis is different from place to place inside the specimen. (4) The conjugate slip system was activated, although the Schmid factor of the slip system was still smaller than that of the primary slip system. This suggested that the activation of the conjugate slip system, i.e., the onset of stage II, is not simply determined by the magnitude of the Schmid factor, assuming the stress state is uniaxial along the tensile direction. (5) Kink bands were formed at the onset of stage II, and it was considered that the formation of kink bands is a trigger for the activation of the conjugate slip system.

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REFERENCES


## Table 4

| Point-to-origin misorientation measured along a line in Fig. 8(b). |
|---------------------------------|----------------|
| Measured between               | Relative misorientation (degree) |
| (I) and (II)                   | 6.0 |
| (I1) and (III)                 | 2.8 |
| (III) and (IV)                 | 1.1 |
| (IV) and (V)                   | 1.7 |
| (V) and (VI)                   | 3.8 |
| (VI) and (VII)                 | 4.4 |

[121], so the formation of the kink boundaries causes crystal rotation with respect to the [121] direction, as shown in Fig. 8(e). It should be noted here that clear kink bands were observed at the end of stage II. Kink bands are one of the deformation bands caused by non-uniform slip and are sets of edge dislocations arrayed perpendicular to the activated slip system of [111]{110}, i.e., [111]{101} in this study. It was found that the tensile axis direction (A2) is still within the standard triangle, even at the end of stage II, as shown in Figs. 5(f) and (g). As shown in Fig. 5 and Table 3, the conjugate slip system was activated, though the Schmid factor of the conjugate slip system was still smaller than that of the primary slip system. It is to be noted that the onset of the conjugate slip system, i.e., the onset of stage II, is not determined simply by the magnitude of the Schmid factor.

Takamura et al. investigated kink bands formed in a Cu single crystal and pointed out the formation of kink bands as a trigger of stage II. The onset of stage II can be considered as follows with the formation of kink bands. First, plastic deformation appears homogeneously with the primary slip system in stage I. As stage I continues, the effect of the constraint on plastic deformation due to the clamps used to hold the specimen becomes nonnegligible. In order to relax the stress field caused by this constraint, dislocations that introduce a bending moment into the specimen are introduced. As the macroscopic deformation continues, dislocations with screw components leave the specimen and then rows of edge dislocations form a wall perpendicular to the slip direction, as shown in Fig. 8(e), resulting in the formation of kink bands. Then, a further bending moment is applied to the specimen. The moment induces tri-axial stress around the kink-bands, leading to an increase in the resolved shear stress on the secondary slip system to promote the activation of the conjugate slip. Several kink bands are formed inside the gauge section, suggesting that conjugate slips can also be activated in the entire specimen, which leads to stage II in the s-s curve. It is expected that the destitution of resolved shear stress should be calculated taking into account the formation of kink bands, which helps to investigate the activation of conjugate slip systems. It should also be noted that the formation of kink bands is the key to the transition from stage I to stage II in the work-hardening process.

## 5. Conclusion

Silicon single crystals were tensile deformed along the [110] direction at high temperatures. The following conclusions were obtained:

1. Silicon crystals showed three clear stages in tensile tests.
2. The strain at the onset of both stage II and stage III decreased with temperature, while work-hardening rates in stages II and III were not sensitive to temperature.
3. The direction of the tensile axis rotated toward [112] during the tensile tests. The crystallographic orientation of tensile axis was dispersed. This indicated that the crystal direction parallel to the tensile axis is different from place to place inside the specimen.
4. The conjugate slip system was activated, although the Schmid factor of the slip system was still smaller than that of the primary slip system. This suggested that the activation of the conjugate slip system, i.e., the onset of stage II, is not simply determined by the magnitude of the Schmid factor, assuming the stress state is uniaxial along the tensile direction.
5. Kink bands were formed at the onset of stage II, and it was considered that the formation of kink bands is a trigger for the activation of the conjugate slip system.