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In situ TEM observations of plastic deformation in quartz crystals

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Abstract With in situ nanocompression experiments in a transmission electron microscope, we investigated plastic deformation in natural quartz crystals and observed both dislocation plasticity as well as mechanical twinning. Through this experimental method, we are able to provide direct evidence of Dauphiné twin nucleation and could measure the intrinsic twinning stress. The twinning phenomena appear to include a memory effect, where the same twin can reappear upon successive loading and unloading events. The data provide insight into this twin generation mechanism and can be used as a benchmark for the use of twins in quartz for paleopiezometry. Together, the observation of room-temperature dislocation plasticity and reversible twinning adds new insight into the extensive field of quartz plasticity and demonstrates the usefulness of small-scale testing techniques for mineral physics.

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Keywords Quartz · Dauphiné twinning · Dislocations · Amorphization · In situ compression · Transmission electron microscopy

Introduction

Quartz is the third most prevalent mineral in the Earth's crust and thought to be a controlling component of plastic deformation in the continental crust (e.g., Ronov and Yaroshevsky 1969; Buergermann and Dresen 2008). It is also widely used as a piezoelectric material as an oscillator in clocks, in microbalances and as actuators (e.g., Bunde et al. 1998; Edwards et al. 1997; Tichy et al. 2010). Natural quartz crystals often contain Dauphiné growth twins. Mechanical Dauphiné twinning occurs geometrically by a 180° rotation around the c-axis that is achieved by a small distortion of bonding in the crystal structure (Schubnikow and Zinserling 1932; Hahn and Klapper 2003). This behavior is a result of the high-elastic anisotropy in quartz, where the direction close to perpendicular to the negative rhomb $\{\bar{1}011\}$ is almost twice as stiff as one perpendicular to the positive rhomb $\{10\bar{1}1\}$ (McSkimin et al. 1965). Compression perpendicular to the negative rhomb flips the orientation to the positive rhomb with minimal macroscopic strain. This conversion is most easily activated at intermediate temperatures below the hexagonal–trigonal phase transition at 573 °C (e.g., Keith and Tuttle 1952; Van Tendeloo et al. 1976; Barber and Wenk 1991).

Mechanical twinning in quartz was first discovered by Schubnikow (1930) by impacting a pin on a crystal with a hammer stroke. Since then, the relevance of this mechanism has been recognized by both materials science where stress is used to remove twins from piezocrystals (Wooster and Wooster 1946) and by geosciences where twins can be

used as paleopiezometers in naturally deformed rocks (Tullis 1970). In materials science, untwinned quartz crystals are preferred for piezoelectric applications because twins reduce their quality. In general, high-quality synthetic single crystals are used for such purpose, but twin structures can be controlled by applying stress (Thomas and Wooster 1951). Recently, characteristic patterning of stress-induced twins in quartz was investigated for nonlinear optical applications (Kurimura et al. 2011). In structural geology, quartz twinning has been used to determine the deformation history of rocks. This is particularly significant in highly stressed conditions, such as meteorite impacts (Trepmann and Spray 2005) and earthquake ruptures (Wenk et al. 2011), where twins have been identified with electron microscopy. In polycrystalline quartz aggregates, twinning has also been induced experimentally (Tullis 1970; Tullis and Tullis 1972; Wenk et al. 2006, 2007).

Important questions about twinning in quartz remain. For instance, how does twinning depend on crystal orientation and what are the necessary stresses to induce twinning? Compression experiments have suggested that twinning becomes active at 200–300 MPa for most favorably oriented crystals (Markgraaff 1986; Wenk et al. 2007), but precise measurement of the local stress to nucleate twins has not been reported. Here, we directly observe and measure the twinning behavior in precisely oriented quartz crystals using a quantitative mechanical deformation stage inside a transmission electron microscope (TEM) (Minor et al. 2011; Shan et al. 2008). This technique has been applied successfully to study mechanical twinning in metals where room-temperature twinning was expected (Ye et al. 2011; Yu et al. 2012). We use this new technique to measure the stress required for twinning in quartz and discuss the twinning mechanism in light of our in situ observations.

Through this pilot study, we have concentrated on the exciting observations of reversible twinning, but also observed dislocation plasticity during deformation and amorphization from the electron beam. Further work is needed to investigate the character of defects and the role of radiation damage from the electron beam.

Experimental

A plate with the largest faces parallel to the $\{10\bar{1}1\}$ plane was cut from a large natural quartz crystal and then mechanically ground to a thickness of about 30 μm . It was confirmed that the plate is a single crystal by electron backscatter diffraction (EBSD). The sample was mounted on a half-moon TEM grid by epoxy glue. Amorphous carbon was deposited on both the sides of the sample to minimize charging problems by ion beam during sample milling,

using the focused ion beam (FIB) technique. Nanopillars were fabricated by FIB operated at 30 kV with a Ga^+ ion source (Strata DB 235, FEI). The long axis of the pillars was parallel to the $\{10\bar{1}1\}$ plane and $\sim 13^\circ$ inclined from normal to the $\{10\bar{1}1\}$ plane (The angle between the $\{10\bar{1}1\}$ and $\{1011\}$ planes is 103.6° in α -quartz). Note that an error of several degrees is likely due to misalignment during sample preparation. One side of the sample was further coated by thin amorphous carbon to prevent charging during TEM observation.

In situ compression testing was carried out on nanopillars in a TEM operated at 300 kV (JEM-3010, JEOL) with a TEM indentation holder with a diamond flat punch (PI-95, Hysitron). In this holder, the load axis is designed to be parallel to the sample rotation axis (about $\pm 30^\circ$ rotation in the JEM-3010). The holder only has one tilt axis; therefore, it is not possible to perform tilt experiments to obtain the ideal two-beam condition to determine Burgers vectors directly in the sample. The tests were executed under displacement control of the loading rate at 1–3 nm/s. Using this system, force and displacement of the flat punch were recorded at an interval of 5 ms. The force was converted to engineering stress for data analysis. In this paper, the engineering stress was defined as the measured force divided by the area of the pillar top, and so the effective stress becomes smaller at the lower part of the pillar because the shape of a pillar prepared by FIB is typically tapered. During compression testing, structural change of the pillars was recorded as a movie at 10–15 frames per second by a CCD camera in the TEM (SC200D, Gatan). Movies are provided as supplementary material (Movie S1–S4).

Results and discussion

Dauphiné twinning

Figure 1 illustrates twinning of a quartz pillar by in situ compression testing. Figure 1a shows a TEM image of the pillar before compression testing taken under a $g_{\bar{1}2\bar{1}1}$ two-beam condition (imaging condition will be discussed later). The diameter of the pillar is ~ 470 nm at the top, and the taper angle is $\sim 4^\circ$. No obvious defect contrast can be seen (Note that $g_{\bar{1}2\bar{1}1}$ may not visualize defects clearly because its structure factor is small). Still images captured from the video recorded during the compression testing (Movie S1) are shown in Fig. 1b–g, where the loading rate was 3 nm/s and the unloading rate was about 35 nm/s. The dashed lines put on the images highlight the characteristic defect contrasts (The contrasts behind the dashed lines can be seen in Movie S1 and Fig. 2). Figure 1h shows a TEM image of the pillar after compression testing. A plot of engineering stress versus displacement of the test is shown in Fig. 1i. Labels

Fig. 1 Deformation of a quartz pillar by twinning. The characteristic twin features in the images are highlighted by the dashed lines. **a** A bright-field TEM image using $g_{\{12\bar{1}1\}}$ before compression and the corresponding diffraction pattern aligned to the image, where the $\{12\bar{1}1\}$ plane is almost parallel to the incident direction and the $\{10\bar{1}1\}$ plane is a few degrees off to the incident direction or to the plane of the pillar top. **b–e** Still images captured from a movie recorded upon loading (Movie S1). Two different twins nucleated from the points indicated by arrows 1 and 2 in **b**, **d**. **e–g** Still images captured from a movie recorded upon unloading. The second twin shrank into arrow 3 in **f**. The first twin also shrank into the left-hand side of the pillar. **h** A TEM image after compression. The characteristic twin features are disappeared. **i** A plot of engineering stress versus displacement taken through the test. The labels **b–g** correspond to the captured image with the same notations

Fig. 2 Retwinning by the second compression (Movie S2). **a** TEM images taken during the first and the second compression at the same stress level are paired. **b** Plots of engineering stress versus displacement of the first compression and the second compression. The horizontal lines indicate the stresses corresponding to **a**

(b–g) on the plot indicate the position of the captured images. A significant strain burst can be seen at point (b). It corresponds to the appearance of a dark contrast feature of about 400 nm in length (arrow 1 in Fig. 1b) and relatively weak-curved contrasts over the pillar, which would correspond to dislocations. The dark contrast grows upon further loading (Fig. 1c) and evolves into a knife-shaped feature in Fig. 1d. An additional band of dark contrast (arrow 2 in Fig. 1d) leads to a second knife-shaped feature as shown in Fig. 1e, partially overlapping the first one. Upon unloading (Fig. 1e–h), both features progressively disappear. At first, the second feature shrinks to the position pointed out by the arrow 3 in Fig. 1f, and then the first feature shrinks toward the left-hand side of the pillar (Fig. 1g) until they both completely disappear (Fig. 1h). The complete disappearance of

the defect features demonstrates the usefulness of the in situ technique.

The strain burst associated with the point (b) in Fig. 1 indicates the formation and movement of the initial defect nucleus. At this moment, the pillar itself demonstrates almost no bending; namely, the crystal orientation should be the same. It is unlikely that this contrast corresponds to some pre-existing defects visualized by crystal orientation change. In addition, we confirmed a similar evolution of knife-shaped contrast in different pillars induced by in situ compression testing. Indeed, the characteristic deformation feature observed during nanocompression tests was a planar, knife-shaped contrast with a sharp corner. In addition, the two knife-shaped contrast features shown in Fig. 1 separately nucleated, propagated and overlapped. Thus, it is

reasonable to consider that the contrast changes observed in Fig. 1 correspond to the evolution of two individual defects. Further, the disappearance of the features upon unloading suggests that the features are not due to dislocation plasticity (section “Plastic deformation by dislocation slip”). Instead, we attribute these features to Dauphiné twins.

Dauphiné twinning in α -quartz is geometrically described by a twofold rotation about the [0001] axis. This twin operation belongs to the point group of the hexagonal lattice but not to that of the trigonal crystal. It is referred to as twinning by merohedry (Hahn and Klapper 2003). This relationship means that the lattices of the twinned individuals completely overlap, and therefore, the positions of the reflections in reciprocal space coincide with those of the perfect crystal. Although the reflection intensities differ due to the symmetry by the twinning, it is difficult to identify them by standard selected-area diffraction in the TEM (Jacob and Cordier 2010). For TEM imaging, Dauphiné twins can be identified under some g conditions, which can be evaluated from the ratio of the scattering amplitudes of left-handed quartz to Dauphiné twin $|F_{\text{LH}}|/|F_{\text{DT}}|$ and the phase difference α (McLaren and Phakey 1969). The reflection of $g_{0\bar{1}11}$ ($|F_{\text{LH}}|/|F_{\text{DT}}| = 1.55$, $\alpha = 0^\circ$) is a candidate to visualize Dauphiné twin and is accessible within the tilting limit in our experiment. However, we could not find a two-beam condition with $g_{0\bar{1}11}$, and so we selected a reflection of $g_{\bar{1}2\bar{1}1}$ ($|F_{\text{LH}}|/|F_{\text{DT}}| = 1$ and $\alpha = 161^\circ$) under a two-beam condition for imaging of the Dauphiné twins as shown in Fig. 1. The contrast exhibited in the pillar is interpreted as α -fringes at the twin boundary, although no stripe contrast is seen. This may be related to the three-dimensional shape of the Dauphiné twins in the pillar, but further analysis is needed to interpret it completely.

After the strain burst (b in Fig. 1i), the slope of the engineering stress plot decreases from 29.1 to 22.4 MPa/nm and is almost constant (constant slope from c to d in Fig. 1i). The in situ observation shows that this decrease of slope is a result of the propagation of the Dauphiné twins and the dislocations. The contribution of plasticity due to the dislocations would be small because only a few dislocations were generated. Thus, the decrease of slope indicates that the deformation resistance of the pillar decreased as mediated by Dauphiné twinning. This is consistent with the stiffest direction flipping to the softest direction by Dauphiné twinning, as expected from the orientation relationship in our experiment.

The local minimum stresses at the strain burst were about 970 MPa, and the diameter of the pillar at the nucleation point of the first twin was ~530 nm, which is ~13 % larger than that of the pillar top. Thus, the nominal stress for the twinning is calculated to be 760 MPa. It was reported that Dauphiné twins are formed by a nominal

average stress of about 200 MPa in polycrystalline quartz (Wenk et al. 2007), but the local stress for twinning has never before been reported. In addition, the twins continued to grow until the nominal stress reached 3,320 MPa, and then they disappeared with unload. This implies that the Dauphiné twins created here are essentially unstable without an applied stress.

The twins formed in the middle of the pillar, where the stress should be lower than that of the top region because of its tapered shape. Since the pillar does not seem to be bending when the first twin nucleated, stress concentrations are unlikely to occur. Note that there is no strain burst associated with the formation of the second twin. Therefore, it is presumed that the strain burst is due to a change of the deformation resistance and not the nucleation of a Dauphiné twin itself. Importantly, we did not observe any defect contrast at the twin nucleation points when the twins were fully extended (Fig. 1e), implying that the source of Dauphiné twins is not sessile, as might be considered by dislocation source models such as the pole mechanism that require the extension of a sessile dislocation (Cottrell and Bilby 1951; Christian and Mahajan 1995). Further, a dislocation source model would not result in the movement of the twin away from the nucleation point, as seen here. The lack of dislocations involved in the twinning mechanism is reasonable since it is known that Dauphiné twinning accommodates no permanent strain (Hahn and Klapper 2003; Tullis and Tullis 1972), which would be inconsistent with a dislocation-based model of the twinning mechanism.

After unloading, the twinned region of the pillar seems to be completely recovered (Fig. 2h). In order to observe whether the pillar would deform in the same manner again, we recompressed the pillar under the same conditions (Movie S2). Figure 2 shows the same pillar under compression a second time with the results from the first compression also shown for reference. Figure 2a shows still images captured from the movies of the first and the second compression, where the image pairs correspond to the same engineering stress in each compression test. The deformation behavior during each compression appears to be quite similar. In particular, the knife-shaped twin features in the image pairs of 2,000 and 4,000 MPa resemble each other closely, indicating that the twin propagation is remarkably repeatable, in effect a retwinning of the same twin due to a memory effect. Thus, the knife shape must be the most energetically efficient under the compression with the present crystal orientation and the form of the tapered pillar. The engineering stress versus displacement plots of the first and the second compression are shown in Fig. 2b. The horizontal lines correspond to the stress levels of the image pairs in Fig. 2a. Interestingly, the plot of the second compression has no strain burst and its slope is almost the same to that of the first compression after the strain

Fig. 3 Deformation of a pillar by dislocation slip. **a** A TEM image before compression and a corresponding diffraction pattern aligned to the image. A projection of an adjacent protrusion of the crystal can be seen near the bottom of the pillar. The $\{1101\}$ and $\{1211\}$ planes are almost parallel to the incident direction and the $\{1011\}$ plane is a few degrees off to the incident direction or to the plane of the pillar

top. **b–d** Still images captured from a movie recorded during the test (Movie S3). **e** A TEM image after compression and the corresponding diffraction pattern. **f** A plot of engineering stress versus displacement taken through the test. The labels **b–d** correspond to the captured image with the same notations. The *dashed lines* correspond to the slope of elastic response of the pillar

burst, suggesting that the twin nucleation processes differs between the first and the second compression. Presumably, small twin nuclei could have remained in the pillar after unloading at the end of the first experiment, and thus when reloaded the same twin structures are formed without a strain burst. These results imply that quartz crystals can contain a memory of their Dauphiné twinning history.

The origin of the twin nuclei most likely comes from the free surface or defects in the crystal. It is known that quartz is sensitive to the electron beam and an amorphous phase is easily formed from free surfaces by electron irradiation (Das and Mitchell 1974). Amorphization of the pillar during the in situ TEM experiment is not negligible (we used a low-beam current condition to minimize any amorphization. cf. section “Amorphization”). The observation that the twin structure is repeatable leads to the conclusion that the nuclei must have been located within the crystal, since it is unlikely that the atomic configuration of the surfaces of the pillar would have been preserved perfectly under electron irradiation.

Plastic deformation by dislocation slip

Figure 3 shows an example of deformation by dislocation plasticity. Figure 3a shows a TEM image of the pillar

before the compression testing, the diameter of the pillar is ~ 300 nm at the head and the taper angle is $\sim 5^\circ$. The dark small region at the bottom right is a shadow from an adjacent protrusion of the crystal. Pre-existing dislocations in the starting material (Ardell et al. 1974) can be seen mid-way down the pillar but no dislocations are apparent at the top of the pillar. Still images captured from the video recorded during the compression testing (Movie S3) are shown in Fig. 3b–d. At the beginning of the compression experiment (Fig. 3b), dislocations can be seen to nucleate from the contact surface at the top of the pillar. As deformation proceeds, strain contrast can be seen throughout the pillar (Fig. 3c) indicating that shearing has begun to lead to off-axial strains. In Fig. 3d, the pillar has sheared near the middle of the pillar after the pre-existing dislocations were activated. Figure 3e shows a TEM image of the pillar after the compression testing, where a large shape change has occurred through dislocation plasticity. A plot of engineering stress versus displacement of the test is shown in Fig. 3f, and Labels (b–d) on the plot correspond to Fig. 3b–d, respectively. The slope of the plot is gentle until displacement of 52 nm. This may be due to local plastic deformation of a burr or an amorphous phase by FIB milling on the pillar top. The linear part of the plot after 52 nm substantially corresponds to an elastic region,

where its slope is indicated by the dashed line in Fig. 3f. The slope of the plot slightly decreases after (b), with dislocation contrast exhibited within about 300 nm from the pillar top as seen in Fig. 3. This indicates yielding of the pillar, where the stress of the part having dislocations is estimated to be in the range from 1,110 to 1,530 MPa after considering the tapered shape. Assuming a Schmid factor of 0.5, this stress range corresponds to a shear stress of 555–765 MPa, which is about 1/60–1/50 of the shear modulus of α -quartz, 33.6 GPa (McSkimin et al. 1965). This high shear stress may be due to a size effect. In general, metallic crystals show a very high yield strength at small scales (Uchic et al. 2004). α -Quartz might also show similar behavior but it has not been investigated before. It should also be mentioned that it has been reported that quartz weakens through the dissolution of water (Griggs and Blacic 1965). This phenomenon shows a critical weakening temperature depending on hydrogen concentration and does not appear at lower than 200 °C, even in highly “wet” quartz (H/Si = 8,800 ppm) (Griggs 1967). In our experiment, the compression testing was performed at room temperature, and a heating effect by electron irradiation can be negligible as discussed later. Therefore, we do not consider the phenomenon of water weakening in quartz to be important in regard to our room-temperature experiments.

The plastic strain of the pillar can be estimated from the displacement. In Fig. 3, the left dashed line is an approximate line corresponding to the elastic response of the pillar, and the right dashed line has the same slope to the left one. From the difference between the intersection points of two dashed lines to the x-axis, the maximum displacement by plastic deformation is estimated to be 134 nm. The dislocations were formed within the top ~800 nm of the pillar. Using this length as the initial pillar length, the plastic strain is estimated to be 17 %.

The plastic strain of 17 % is large because ceramics and particularly quartz are generally brittle at room temperature and low pressure (vacuum). At small scales, however, it has been shown that room-temperature ductility is possible in covalently bonded ceramics that do not show ductile behavior in bulk samples (Minor et al. 2005; Ge et al. 2006; Deneen et al. 2006; Han et al. 2007; Östlund et al. 2009, 2011; Jian et al. 2012). The effect of source truncation due to small volumes and the nearby surfaces leads to easy dislocation nucleation at high stresses, which in turn can lead to large strains through dislocation plasticity. In addition, it is important to consider the effect of electron irradiation, which is known to cause damage and even amorphization of quartz, which will be discussed below. As seen in Fig. 3e, the head of the pillar is slightly squashed. This is attributed to deformation of an amorphous phase formed by FIB and electron irradiation. The amorphous phase forms on the surfaces of the pillar, which can be seen as a relatively bright contrast

in Fig. 3. Comparing Fig. 3a with Fig. 3e, we found that the amorphous phase thickened from 38 nm to 45 nm during the test. The difference, 7 nm, is only ~5 % of the total displacement of the pillar, and thus, it can be said that the amorphous phase itself did not contribute much for the plastic deformation. However, since the electron irradiation can lead to bond breaking in the quartz structure, it may enhance dislocation activity and facilitate plastic deformation.

Amorphization

Figure 4 illustrates amorphization of a pillar during compression testing. Figure 4a shows a TEM image of the pillar before compression testing. The diameter of the pillar is ~270 nm at the top, and the taper angle is ~4°. The pillar is covered by an about 30-nm-thick amorphous layer due to FIB fabrication. Still images captured from the video recorded during the compression testing (Movie S4) are shown in Fig. 4b, c. A plot of engineering stress versus displacement of the test is shown in Fig. 4f, and (b, c) on the plot correspond to Fig. 4b, c, respectively. The deformation of the pillar proceeds with squashing of the pillar head (Fig. 4c). Upon unloading, the force is negative after displacement of ~270 nm, indicating that the pillar was elongated with pulling back the flat punch since they adhered to each other. Figure 4d, e shows TEM images taken in the stage of pulling back the flat punch by manual operation. The pillar was considerably elongated to ~2.3 μm (Note that the length was measured within Fig. 4e, because the exact length of the pillar is difficult to estimate in the present case). Since the initial length of this part of the pillar was ~1.9 μm , the elongation strain is estimated to be 21 %. It is found from the diffraction patterns in Fig. 4e that the elongated part is an amorphous phase, whereas the body of the pillar has some crystalline portions, also indicated by contrast. The thickness of the amorphous layer covering the body of the pillar has increased to 70–80 nm. Amorphization proceeded from the surfaces of the pillar by electron irradiation. In this process, a large portion of plastic deformation is due to the amorphous phase formed by electron irradiation.

The temperature increase in a sample due to electron irradiation has been discussed for amorphous silica by Zheng et al. (2010). The energy transfer rate which the sample obtains from the electron beam at an energy density rate is written as $H = QJ$, where Q is the stopping power of an electron (the average energy loss of an electron per unit length) and J is the electron current density. The current density of the electron beam was $5.8 \times 10^5 \text{ pAcm}^{-2}$ in the experiment of twin nucleation (Figs. 1, 2). Stopping power is written as $Q = Q_c + Q_r$, where Q_c is the collision stopping power and Q_r is the radiative stopping power. In the present case, Q_c and Q_r are estimated to be 0.52 and $2.16 \times 10^{-3} \text{ eV nm}^{-1}$, respectively (Berger et al. 2005),

Fig. 4 Amorphization of a pillar during compression. **a** A TEM image before compression. **b–d** Still images captured from a movie recorded during the test (Movie S4). **e** A TEM image after compression

where the energy of electrons $E = 300$ keV and the mass density of quartz $\rho = 2.65 \times 10^3 \text{ kg m}^{-3}$ were used. Using the estimated values of Q and J , the energy transfer rate is evaluated to be $H = 3.03 \times 10^6 \text{ W m}^{-3}$. In case of an isolated sample, the heating rate is $H/\rho C = 1.5 \text{ K s}^{-1}$, where $C = 0.75 \text{ J g}^{-1} \text{ K}^{-1}$ is the specific heat of quartz.

The heat dissipates because of thermal conduction through the sample. In an equilibrium state, the temperature field in the sample is satisfied as the following relationship:

$$k \nabla_r^2 T = -H, \quad (1)$$

where k is the thermal conductivity. The thermal conductivity of quartz is $k \sim 10 \text{ W m}^{-1} \text{ K}^{-1}$ (Kanamori et al. 1968). Substituting the H estimated above and the thermal conductivity k into Eq. (1), the temperature field is $\nabla_r^2 T = 4 \times 10^6 \text{ K m}^{-2}$. Since the size of the pillars is of the order of μm , the temperature difference is only $\sim 10^{-6} \text{ K}$. Also, the heat dissipation occurs through the interface between the sample and the diamond flat punch during indentation. The thermal conductivity of diamond $k \sim 3,000 \text{ W m}^{-1} \text{ K}^{-1}$ (Anthony et al. 1990) is much higher than that of quartz. Thus, the temperature difference between the sample and the flat punch should be much

lower than that in the quartz pillar, unless the thermal conductance at the interface is very low. Therefore, the temperature increase of the pillar owing to electron irradiation is considered negligible.

In the compression testing shown in Fig. 4, the pillar amorphized and elongated considerably where the current density of the electron beam was roughly $1 \times 10^7 \text{ pA cm}^{-2}$, which is about 20 times larger than that in the twinning case (Figs. 1, 2). This value, however, is still too low to increase the temperature of the pillar appreciably. Amorphous silica is known to be highly deformable under the electron beam, and this is considered to be due to not a thermal effect but incremental bond-switching events among atoms by electron irradiation (Zheng et al. 2010). In our experiment, the situation should be similar—electron irradiation brought about the formation of an amorphous phase from the quartz pillar surfaces, and it assisted the elongation of the pillar as well.

Conclusion

Our exploratory in situ TEM deformation experiments provide direct evidence of a twinning, detwinning and

retwinning phenomenon in quartz at room temperature. The results imply that mechanical twins can be formed during deformation from a non-sessile type nucleus but are unstable and will detwin upon unloading. Interestingly, once a twin forms, the deformation resistance changes, and there is a possibility of retwinning from a memory effect. We also observed at room-temperature large plasticity of quartz due to dislocation slip and amorphization. Both appear to be enhanced by electron irradiation in a TEM. The preliminary results call for future systematic investigations with similar techniques at a wide range of temperatures and crystal orientations.

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