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Another origin of yield drop behavior in sapphire deformed via basal slip: Recombination of climb-dissociated partial dislocations



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ABSTRACT

It is demonstrated from repeated loading-unloading high-temperature deformation tests of sapphire crystals deformed via basal slip that the deformation stresses go back to the same level as just before unloading after a small yield drop is observed during the re-loading process. This phenomenon suggests that basal dislocations that work as primary slip system regain the ability of glide motion even after they are dissociated into two partial dislocations via self-climb. That is, the climb-dissociated partials in basal dislocations introduced by previous deformation recombine onto the same basal plane so that they can glide again, resulting in the yield drop.

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Single crystals with low initial dislocation density often experience a significant yield drop when deformed at a constant strain-rate. In general, two mechanisms are used to explain the reason for the yield drop behavior of a single crystal, (i) breaking away from a Cottrell atmosphere [1] and (ii) the Johnston-Gilman mechanism [1]. The Cottrell atmosphere assumes that mobile point defects (often solute atoms) are attracted to the strain field surrounding stationary dislocations and that such dislocations need an extra shear stress to initiate dislocation motion by breaking away from these point defects. After the dislocations move away from the point defects, the shear stress required for dislocation motion is reduced, resulting in a yield drop. In case of the Johnston-Gilman mechanism, on the other hand, rapid multiplication of dislocations at the initial stage of plastic deformation allows the dislocations themselves to move more slowly as the strain rate is proportional to both the mobile dislocation density and the dislocation velocity, also resulting in a yield drop after dislocation motion has been initiated.

Sapphire crystals (α -Al₂O₃ single crystals) exhibit a large yield drop at the initial stage of plastic deformation when deformed by basal slip at elevated temperatures (T > 1200 °C) [2–7]. To date, the origins of the yield drop behavior had been believed to be due to one or the other of the above two factors, but it has not been clear whether the

* Corresponding author. E-mail address: anaka@nagoya-u.jp (A. Nakamura). phenomenon is due to breaking away from a Cottrell atmosphere or due to rapid dislocation multiplication after the onset of dislocation motion. Sapphire is one of the most widely used oxide crystals because it has the superior stability and high strength as a transparent structural material under wide-range temperatures. Therefore, to understand the yield drop behavior of sapphire crystals deformed by basal slip at elevated temperatures is of broad interest and of great importance.

In recent years, the core structure of dislocations has successfully been observed at an atomistic level due to the advancement of transmission electron microscopy (TEM) techniques [8–12]. The dislocations that take part in basal slip in sapphire, which is known as the primary slip system at elevated temperatures, have been imaged using high-resolution TEM (HRTEM), and have been shown to dissociate into two partial dislocations via self-climb on a prism plane (i.e., a plane normal to the glide plane) and have a separation distance of about 4 nm [8]. In this case, the climb-dissociated basal dislocations cannot glide as they have a stacking fault on a prism plane, which is normal to the basal plane, unless they recombined onto the same basal plane being the glide plane. Moreover, if the core structure of dislocations recovers to regain the ability of dislocation motion via glide, this recovery process would have to take some time as it involves short range pipe-diffusion and thus may have a potential to bring about a yield drop before dislocation motion via glide can proceed.

In order to investigate whether basal dislocations can move via glide again after initial activation and to evaluate the influence of core

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structure of the dislocations on the yield drop behavior, repeated loading-unloading plastic deformation tests of undoped and Ti-doped sapphire crystals oriented for basal slip were carried out at elevated temperatures after which the core structure of the basal dislocations was directly imaged using HRTEM. In these experiments, plastic deformation tests of Ti-doped crystals were also used to explore the influence of a Cottrell atmosphere as Ti has a tendency to segregate to basal dislocations in sapphire [12–14]. In addition, Ti-doping into sapphire is believed to decrease oxygen vacancy concentration due to the formation of aluminum vacancies as a charge compensating defect when Ti⁴⁺ substitutes for Al³⁺; this would also results in a decrease of the oxygen diffusivity in sapphire [15–16].

Undoped and Ti-doped sapphire crystals grown by Verneuil's method were used in this study; the Ti concentration of the Ti-doped Al₂O₃ crystal was 127.5 ppm by Al site. The size of the specimens prepared for the deformation tests was 5 mm \times 5 mm \times 10 mm and all the surfaces were mechanically polished to a mirror finish with diamond pastes and a colloidal silica. The load axis was set at 45° with respect to both the [0001] and $[2\overline{110}]$ directions. Consequently, the (0001)1/3[2110] basal slip system has the Schmid factor of 0.5. This shape and crystallographic orientation of the specimens have been used in the former research [17]. The specimens were deformed in compression in air at 1400 °C using a constant cross-head speed of 0.006 mm/min, which corresponds to the initial strain-rate of 1.0×10^{-5} /s. After they were deformed up to $\varepsilon = 10\%$, some of the specimens underwent quick unloading after which they were loaded again so that plastic deformation resumed; the temperature and the cross-head speed for the subsequent loading were maintained with respect to the initial loading. This unloading-loading process was carried out two times for each specimen of undoped and Ti-doped sapphire crystals, and thus all the specimens were compressed three times in total. After the deformation tests, introduced dislocations were observed using a conventional TEM (JEOL JEM-2010HC, 200 kV) and HRTEM (JEOL JEM-4010, 400 kV).

Stress-strain curves obtained from the high temperature deformation tests of undoped and Ti-doped sapphire crystals are shown in Fig. 1(a) and (b), respectively. The both specimens exhibited large yield drops at the initial deformation. The Ti-doped sapphire crystal showed rapid work-hardening after the initial yield drop, where the saturation stress is slightly higher than that of the undoped sapphire. This behavior similar to previous studies of undoped and impurity doped sapphire undergoing basal slip [5]. After specimens have undergone initial plastic deformation, they were completely unloaded followed by a subsequent stage of plastic deformation using the same strain-rate and temperature. Three significant results were obtained from this set of deformation experiments shown in Fig. 1(a) and (b). The first finding is that the yield drop behavior persisted even at the re-loading stages after the initial yield drop, although significantly smaller than the initial yield drop. This suggests that the initial yield drop in part is mainly due to multiplication of the dislocations, and that subsequent yield drops appear even after dislocations has multiplied to the extent that a constant strain rate can be sustained. Namely, the initial yield drop behavior in large part can be explained by the Johnston-Gilman mechanism, which requires a multiplication of mobile dislocations so that the strain-rate can be explained by the dislocation density and dislocation velocity. However, the subsequent smaller yield drops observed during the re-loading process after the initial dislocations substructure has developed requires an alternate explanation. The second finding is that similar yield drops takes place for undoped and Ti-doped sapphire crystals. This suggests that breaking away from a Cottrell atmosphere would not be a main origin of the yield drop behavior. This argument can also be confirmed from another experiment, where the dwelltime after unloading was changed (see the Supplementary material on the website). The third finding is that upon re-loading the deformation stresses go back to the same level as before unloading just after the small yield drops. This is an indication that the glide dislocations are immobile and require some time to become mobile, which is consistent



Fig. 1. (a), (b) Stress-strain curves obtained from the high-temperature deformation tests of (a) undoped and (b) Ti-doped sapphire crystals.

with the recombination of climb-dissociated partial dislocations back onto the glide plane. If all the dislocations introduced by the initial deformation become sessile (through conservative self-climb), new basal dislocations have to be generated via dislocation multiplication in order for the plastic deformation to continue. In that case, one would expect that the yield drop observed during the re-loading process would be similar in magnitude to the initial yield drop as more strain is needed to achieve the same stress level as the initial deformation. That is, the dislocations introduced by the initial deformation regain the ability of glide motion during the subsequent deformations.

Typical TEM bright field images of dislocation substructures after the deformation tests of an undoped and a Ti-doped sapphire crystal deformed to a strain of $\varepsilon = 10\%$ are shown in Fig. 2(a) and (b), respectively. It should be noted that the dislocation substructure of sapphire deformed via basal slip depend on the type of impurity doping, and have been discussed in detail by Heuer and co-workers [18–20]. The substructure of Ti-doped sapphire has similar density of dislocations although the density of dislocation loops is larger than that for undoped sapphire [18]. Typical HRTEM images of basal dislocations introduced by the plastic deformation of undoped and Ti-doped sapphire crystals deformed to a strain of $\varepsilon = 10\%$ are shown in Fig. 2(c) and (d), respectively, where (c) and (d) correspond to the images of edge type basal dislocations. It can be seen that the basal dislocations dissociate into two partial dislocations by climb perpendicularly to the glide plane in both undoped and Ti-doped sapphire crystals. The separation distances between the partials in Ti-doped sapphire is almost the same compared as for undoped sapphire, suggesting that the stacking fault energy of the prism plane is similar for the two crystals. It is also likely that the dopants do not significantly affect the core structure of basal dislocations

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