In-situ transmission electron microscopy of partial-dislocation glide in 4H-SiC under electron radiation

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(Received 21 June 2012; accepted 5 July 2012; published online 23 July 2012)

Electron-radiation-enhanced glide of 30°-Si(g) partial dislocations bringing about an expansion/shrinkage of Shockley-type stacking faults in 4H-SiC was observed *in-situ* by transmission electron microscopy. Geometrical kinks on 30°-Si(g) partials did not migrate in the dark, indicating that the kink migration is enhanced by electron irradiation. The direction of the enhanced glide was reversible depending on the irradiation intensity, which can be interpreted in terms of a sign reversal of the driving force originating in the effective stacking fault energy variable with the irradiation intensity.
 [http://dx.doi.org/10.1063/1.4737938]

Silicon carbide (SiC) is a promising material suited for power devices due to its superior outstanding properties such as high breakdown voltage and low power loss. Despite the outstanding properties, PiN diodes on 4H-SiC degrade under electronic excitation conditions such as forward biasing[1] and optical excitation.[2] This degradation process is due to the expansion of Shockley-type stacking faults (SFs) bound by 30° partial dislocations terminated with Si atoms (so-called 30°-Si(g) partials) and those terminated with carbon atoms (30°-C(g) partials) accompanied with enhancement of the dislocation mobility[3,4] that was revealed by photo- and electro-luminescence imaging. It is shown that only 30°-Si(g) partials are movable during excitation,[5–7] and the activation energy is reduced to about 0.27 eV (Refs. 2 and 3) from the energy without excitation (about 1.3 eV).[8] Since the partials are immobile in the dark at room temperature (RT),[8] it is commonly believed that they can move via the radiation-enhanced dislocation glide (REDG),[9] that is driven by the effective negative formation energy of SFs via the trapping of excitation-induced carriers at the SFs.[10] In recent optical experiments, one of the present authors proposed that the sign reversal of stacking fault energy is brought about by trapping of excitons by the SF.[11,12] This is consistent with the fact that SFs shrink in darkness or in the lack of high density of excitons at temperatures high enough for partials to glide thermally.[13–15] However, the fundamental mechanism of the dislocation glide under electronic excitations has not been fully established due to the lack of experimental knowledge at a microscopic level on the dynamics of the partial dislocations under electronic excitation. In the present work, REDG of 30°-Si(g) partials bringing about an expansion/shrink of Shockley-type SFs is observed *in-situ* by transmission electron microscopy (TEM), using an electron beam for TEM to excite electrons similar to the case of forward biasing and optical excitation. The experimental results provide a proof for that kink migration is enhanced. The direction of the enhanced glide is reversible depending on the electron irradiation intensity, which can be interpreted by the sign reversal of the SF-originated driving force for the enhanced glide.

The sample was an undoped 4H-SiC (with the nitrogen concentration of $1.5 \times 10^{14}\,\mathrm{cm}^{-3}$) film of 17 μm thick homoepitaxially grown on the Si face of a 4H-SiC substrate 8°-offcut towards $[11\bar{2}0]$. The film surface on a small chip of the sample was indented at RT, and fresh dislocations were introduced intentionally. Then, the $(000\bar{1})$ surface of the chip was mechanically polished until the chip was about a few micrometers in thickness. The polished chip was thinned until the chip was sufficiently thin for TEM by mechano-chemical etching with silica nanoparticles, without ion milling, so as to reduce surface defects.[16] Here, it was found that REDG is not observed in thinner specimens, presumably due to surface defects that act as recombination centres. The specimen thickness was therefore chosen about 600–1000 nm even though it was rather thick for ordinary TEM observations. A 120-keV electron beam was used for TEM at RT so as to reduce the introduction of point defects inside specimens, which would act as recombination centres.[17] Actually, dislocations were immobilized when they were irradiated with a small amount of 160-keV electrons exceeding the radiation-damage threshold. A rise in temperature due to electron irradiation in our microscope was considered to be 20–30 K at most.[18] Remnants of silica nanoparticles on a surface were used as markers.

A number of half loops of dislocations on the (0001) basal planes were generated from the edge of an indent. The inner part of each loop exhibited a distinct contrast when the diffraction vector **g** was not normal to [0001], indicating that each loop is consisted of partial dislocations bounding a SF. The dislocation loops were observed to expand via a glide of the partial dislocations under an electron irradiation condition (Figs. 1(a)–1(d)). Since the loop was invisible when $\mathbf{g} = 11\bar{2}0$ (Fig. 1(e)), the Burgers vector **b** was identified to be either parallel or antiparallel to $[\bar{1}100]$. Hence, the loop was consisted of two elongated screw segments bridged by two 30° partial segments in different orientations. The 30° segments exhibited an inside contrast for $\mathbf{g} = 1\bar{1}00$ with $s > 0$ (Fig. 1(f)), indicating that $(\mathbf{g} \cdot \mathbf{b})s < 0$ for the FS/RH

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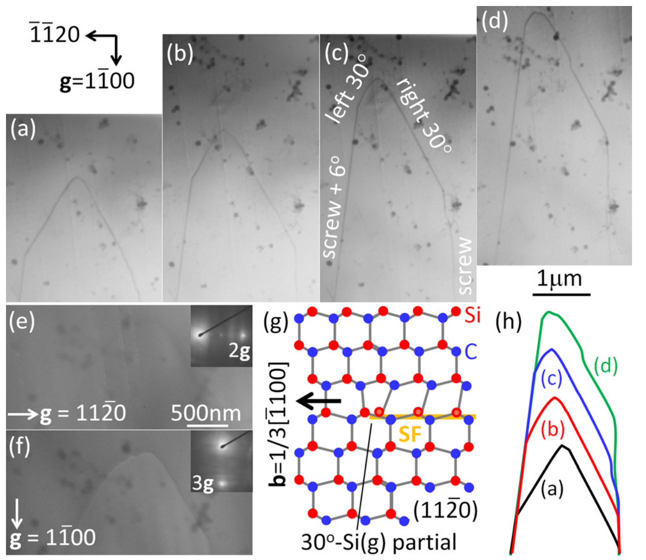

FIG. 1. (a)–(d) Bright-field (BF) TEM images of 30° partial segments after electron irradiation with $I = 5\,\mathrm{A/cm^2}$ ($\mathbf{g} = 1\bar{1}00$). Irradiation time is (a) 0, (b) 60, (c) 120, and (d) 180 s. These snap-shot images were acquired in sequence from (a) to (d) with $I = 3 \times 10^{-2}\,\mathrm{A/cm^2}$. A DF image of the 30° segments taken with $\mathbf{g} =$ (e) $11\bar{2}0$ or (f) $1\bar{1}00$, where the insets indicate the reflection conditions. (g) A structural model of the 30°-Si(g) partials. (h) A sketch of the dislocation line shapes in (a)–(d).

convention;[19] $\mathbf{b} = 1/3[\bar{1}100]$. The segments were therefore 30°-Si(g), the hypothetical core structure of which is shown schematically in Fig. 1(g).

The two 30°-Si(g) partial segments were observed to glide at respectively constant velocities under a uniform beam current (Fig. 1(h)). The glide velocity depended on the beam current density I and became zero when the electron irradiation was ceased (Figs. 2(b) and 2(c)), indicating that the observed glide of the 30°-Si(g) partials represents REDG induced by TEM electron beam irradiation. In contrast, the screw partials stayed immobile under electron irradiation (Figs. 1(a)–1(d)), indicating that screw partials do not exhibit REDG effects at the investigated intensities.

It should be noted that the partial dislocation line of the left screw segment in Figs. 1(a)–1(d) is appreciably tilted by clockwise +6° from the exact direction of b, in contrast to another screw segment on the opposite side of the SF. Such tilted-screw partials were observed commonly on the left side of the 30°-Si(g) partials, though the tilt angle was variable (see Fig. 3).

Another notable fact is that the orientations of 30°-Si(g) partials slightly deviate also clockwise from the Peierls potential valleys indicated with the lines in Fig. 2. The tilted 30°-Si(g) segments contain geometrical kinks of the same sign as schematically shown in Fig. 2(d). Following the terminological convention,[20] we call the geometrical kinks of this sign right kinks and the kinks of opposite sign left kinks hereafter. It must be stressed that the tilted 30°-Si(g) partials stayed immobile in the dark (Figs. 2(b) and 2(c)). This fact indicates that the migration energy of the geometrical right kinks is too high to migrate at RT without electron irradiation. This is in agreement with an *ab-initio* calculation[20] which showed that the migration energy of right and left kinks on 30°-Si(g) partial dislocations in thermo-equilibrium is more than 2 eV if the dislocation core is reconstructed. In other words, the present results strongly suggest that the core of 30°-Si(g) partial dislocations in 4H-SiC is reconstructed at least in the dark.

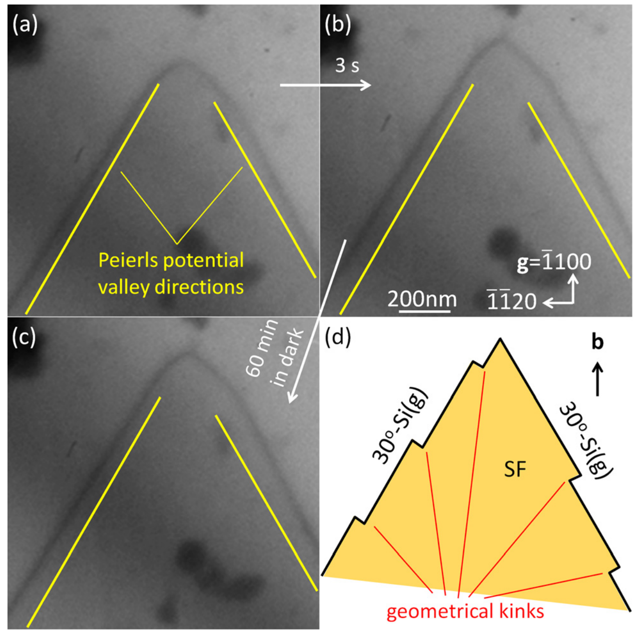

FIG. 2. (a) and (b) Sequential BF-TEM images of tilted-30°-Si(g) partials under electron irradiation for 3 s with $I = 2 \times 10^{-2}\,\mathrm{A/cm^2}$ ($\mathbf{g} = \bar{1}100$). (c) A BF-TEM image of the partials kept in the dark (without electron irradiation) for 60 min after imaging (b). (d) A sketch of the dislocation line shape of the partials.

More important is the fact that the tilted 30°-Si(g) partials become mobile under electron irradiation, which means that the kink migration prohibited in the dark is enhanced under the electron irradiation. Furthermore, this enhanced kink migration can occur reversibly. Fig. 3 shows sequential TEM images of a partial dislocation loop subjected to electron irradiation in different intensities. As the irradiation intensity was increased to some level (in this case $I = 2\,\mathrm{A/cm^2}$), the loop expanded, but when the intensity was decreased, the loop shrunk to recover the loop shape before the intense irradiation. The reversible change of the dislocation loop can be accounted for by the glide of the two 30°-Si(g) partial segments lying at the upper part of the images. Although the partials are immobile in the dark as

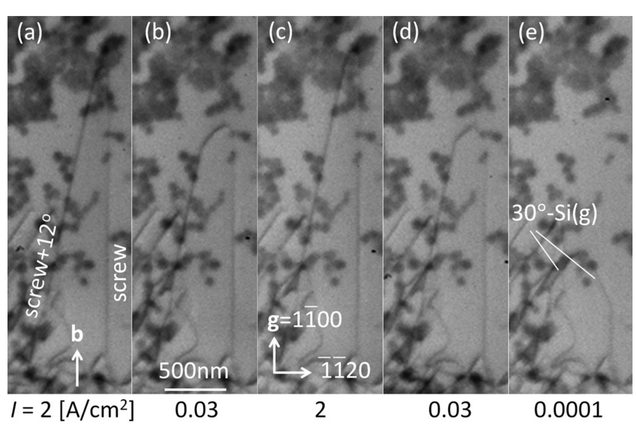

FIG. 3. Reversible change of a dislocation loop under different excitation conditions (BF, $\mathbf{g} = 1\bar{1}00$). The images were acquired in sequence from (a) to (e) with $I = 3 \times 10^{-2}\,\mathrm{A/cm^2}$.

shown above, they maintain mobility as far as the crystal is irradiated with electrons in finite intensities. The glide direction, however, changes depending on the irradiation intensity. At high intensities, the glide occurs to expand the SF, whereas at low intensities, it occurs to shrink the SF. This paradoxical fact can be interpreted in terms of the driving force for the REDG effect: The sign of the driving force arising from the effective formation energy of SF can be reversed by trapping of high-density excitons at the SF,[11] while the REDG effect is induced by electron irradiation to a degree depending on the excitation intensity.

The dominance of right kinks over the left kinks on a 30°-Si(g) partial suggests that the left kinks are more mobile than the right kinks under an electron irradiation condition because less mobile kinks should tend to be accumulated on the dislocation line. This asymmetry in kink mobility under irradiation might be the cause of the difference in the tilt of the screw dislocations that are formed as trails of gliding segments of the 30°-Si(g) partials, though the detailed processes are not known at present.

In conclusion, *in-situ* TEM observations directly proved that the glide of only 30°-Si(g) partial dislocation segments is enhanced by radiation so as to expand/shrink the SF. The glide direction is reversible depending on the irradiation intensity, which can be accounted for by the effective SF energy acting as the driving force for the REDG effect.[11,12] Kinks in the 30°-Si(g) segments did not migrate in the dark, which means that kink migration, as well as kink-pair formation, is enhanced on the 30°-Si(g) segments, as previously suggested.[12]

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