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Dynamical Study of Dislocations and 4H→3C Transformation Induced by Stress in (11-20) 4H-SiC

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Abstract. 4H-SiC samples were bent in compression mode at temperature ranging from 400°C to 700°C. The introduced-defects were identified by Weak Beam (WB) and High Resolution Transmission Electron Microscopy (HRTEM) techniques. They consist of double stacking faults bound by 30° Si(g) partial dislocations whose glide locally transforms the material in its cubic phase. The velocity of partial dislocations was measured after chemical etching of the sample surface. The formation and the expansion of the double stacking faults are discussed.

Introduction

The wide-band-gap semiconductor silicon carbide (SiC), and especially its 4H polytype, is of interest for the fabrication of electronic devices suitable for high-temperature and high-power applications. Recently, a degradation phenomenon associated with the growth of defects was observed in 4H-SiC p-n diodes during forward-bias reliability testing. The responsible defects were identified as single stacking faults (SSF_S) bound by Shockley partial dislocations and their expansion was attributed to the electrical stress [1,2].

Additionally, the formation of double stacking faults (DSF_S) inducing a 4H→3C polytypic transformation was recently observed to occur in highly nitrogen doped 4H-SiC at typical device processing temperatures [3], during further annealing [3,4] or oxidation [5]. DSF_S expansion is ascribed to : i) an internal stress resulting from the lattice mismatch due to either the oxidation or the doping difference between the epilayer and the substrate [5] and ii) the lowering of the energy of highly n doped-crystal through electrons entering Quantum-Well-Like states associated with DSF_S [3,4].

In our work, DSF_S were introduced by external mechanical stress at low temperature in order to measure directly the velocity of partial dislocations bounding them. In this paper, we identified the DSF_S and the created-partial dislocations using Weak Beam and HRTEM techniques. Based on these results and velocity measurements, we attempt to explain both formation and DSF_S expansion.

Experiments

We used (11-20) oriented 4H-SiC nitrogen-doped ($5\times10^{18}cm^{-3}$) wafers provided by Cree-Research. The {0001} glide planes were perpendicular to the sample surface and at 45° from the tension-compression axis X (Fig.1). Dislocations were nucleated by scratching with a diamond tip the sample surface in a direction parallel to X. The samples were deformed by cantilever bending and annealed under stress at temperatures ranging from 400°C to 700°C. The defects emerging at the sample surface were characterized by chemical etching using molten KOH (500°C, 10min).

(11-20) planar views for HRTEM imaging were prepared by mechanical polishing and ion beam milling of the sample back sides. (0001) cross sections for WB investigations were prepared at the tip of chemically etched faults by the Focused Ion Beam technique (CP2M-Paul Cézanne-Marseille). WB and HRTEM were performed at 200 keV using a 200CX Jeol (ONERA-Chatillon) and a 2010-FEG (CP2M-Paul Cézanne -Marseille) respectively.

Results and discussion

Optical images of all deformed and chemically etched samples reveal straight lines parallel to the (0001) basal plane, which correspond to the intersections of macroscopic SF_S with the sample surface. Partial dislocations were nucleated either from the scratch or from the sample edge damaged by the sample cutting. They expand asymmetrically in two opposite directions (a and b) as shown in figure 1. Contrary to SFa_S which develop rapidly away from the scratch (0 – 2.5 mm), SFb_S remain close to the dislocation source (0-100 µm). The velocity v(x) of SFa_S is deduced from the distances *D(x)* covered by the dislocations during the deformation annealing. We measured *D(x)* on images of the etched samples (Fig.1). For the applied stress, since the gliding plane is edge-on and at 45° from the scratch, we take the value $\sigma_r(x)$ where x corresponds to the mid distance *D*/2.

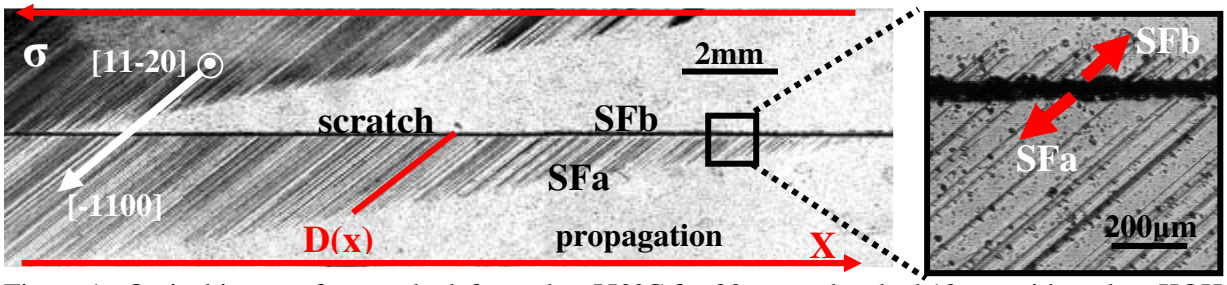

Figure 1 : Optical image of a sample deformed at 550°C for 30 mn and etched 10 mn with molten KOH.

Each macroscopic SF (a or b) consists of two distinct SF_S as revealed by WB images (Fig 2a and 2c). They are bound by a pair of 30° partial dislocations (D1 and D2) aligned along the [-12-10] direction for SFa_S and the [2-1-10] direction for SFb_S. The two partials bounding SFa_S or SFb_S exhibit the same Burgers vector **b** = ± a/3 [-1100].

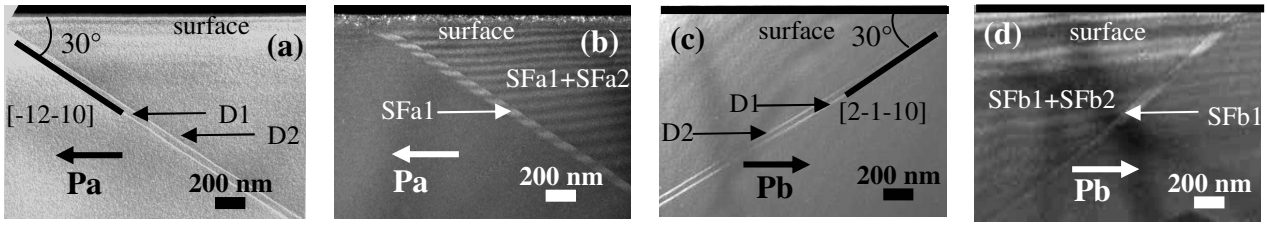

Figure 2 : Cross-sectional WB TEM micrographs of the D1 and D2 30° partial dislocations bounding SFa (a,b) or SFb (c,d). Pa an Pb are the propagation directions of SFa and SFb respectively.

HRTEM images obtained for the two kinds of SF_S shows 3C layers consisting of 6 planes in the 4H-SiC matrix (fig.3a, 3c). Such defects can only be obtained if the two identical partials glide on two adjacent basal planes labelled $GP_{1,2}$ ($GP_{3,4}$) when viewed in the (11-20) plane. The figure 3 shows that the expansion of DSFa (DSFb) produces A-3C (B-3C) stacking when the partials glide in $GP_{1,2}$ ($GP_{3,4}$). Our deformation procedure has thus created DSF_S and not SSF_S. However, in our experiments, the DSF_S expansion in the Pa or Pb direction results in a sole stacking when observed along a well defined crystallographic orientation.

Using an analysis of the HRTEM images [7], we determine the core composition of the partials and the extra half plane location for the two kinds of DSFs. We first localize in the (11-20) plane the projections of the C and Si atomic columns by comparing the contrast of the experimental image (Fig. 3a,3c) with that of DSF_S simulated using the multislice method [6] (Fig. 3b,3d).

In order to derive the core composition of the two 30° partial dislocations for the $DSFa_S$ ($DSFb_S$) we project the structure of the 4H-SiC and of the sheared layers in the (-12-10) plane ((2-1-10) plane) which is perpendicular to the dislocation lines. Knowing the propagation direction Pa (Pb), we rebuild the boundaries in order to obtain the structural unit characteristic of a 30° partial (Fig.4a,4c).

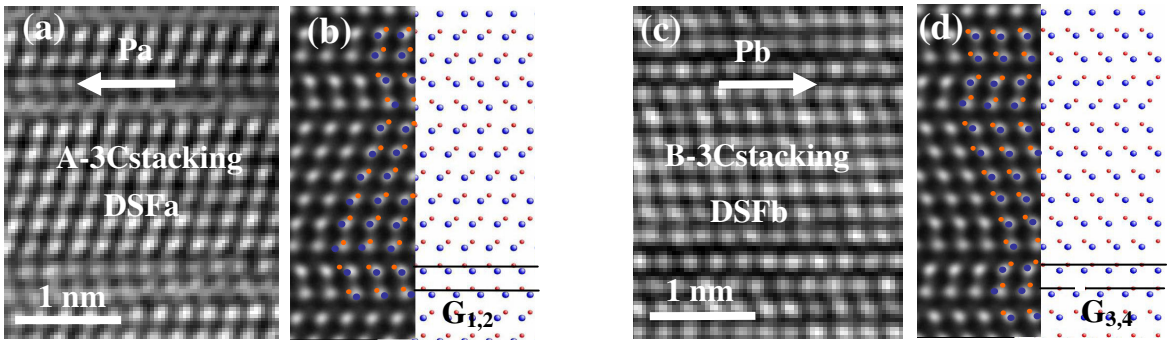

Figure 3 : Projections of DSFa and DSFb in the (11-20) plane: (a,c) HRTEM experimental images; (b,d) contrast simulated using the multislice method and the experimental defocus (Δf=-15nm); the projection of Si and C atomic columns are also shown as big and small circles respectively; G1,2 and G3,4 are the two possible pairs of glide planes .

The reconstruction of $DSFa_S$ ($DSFb_S$) demonstrates that the core of the partials consists of a column of Si atoms, they are thus 30° Si(g). The extra half-planes point downward from the core which induces a sample in compressive (tensile) strain (Fig.4b,4d).

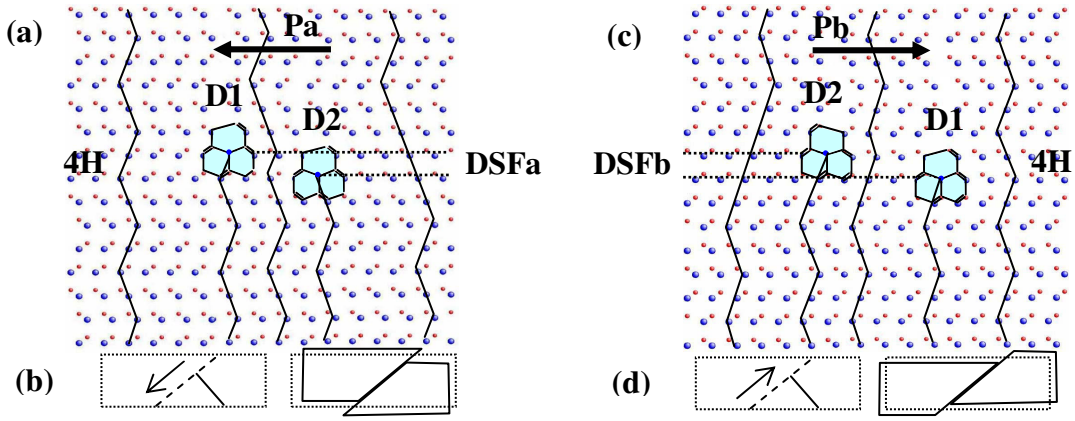

Figure 4 : Projection of the 30° partial dislocation cores in the plane perpendicular to their lines: (a) (-12-10) plane ; (c) (2-1-10) plane. (b,d) : Schematic of the compressive and tensile strain due to the partial dislocation glide.

The $DSFa_S$ which induce a compressive strain rapidly expand as a function of the compressive stress applied on the dislocation sources (scratch) whereas the $DSFb_S$ expansion decreases with the applied stress. Such results are in good agreement with the bending conditions and explain the asymmetry of the deformation observed in our samples. They demonstrate that the applied stress is the main driving force involved in the DSF expansion. However, we have found that the velocity values are higher than expected as compared to silicon [8] in similar stress and temperature ranges (e.g. Fig.5). These high values indicate that additional driving forces have to be considered.

To determine the latter, we analyse the forces applied on the two partials bounding one DSF when the equilibrium distance (d) between the partial lines is reached (Fig.6). These forces are of two kinds: i) a back-stress due to the SF formation (where the energy of formation γ_1 corresponds to a single SF and γ_2 to a second SF nucleated in an adjacent plane) ii) a repulsive force between the two identical bounding partials ($F_R \approx 1.44\times10^{-2} Nm^{-1}$ when $d \approx 60nm$). The values of γ_1 and γ_2 have been calculated in purely intrinsic 4H-SiC ($\gamma_2 \approx 0.31\times10^{-2} Jm^{-2}$, $\gamma_1 \approx 1.84 \times10^{-2} Jm^{-2}$) [9]. If we use these values an additional driving force ($F_{ADD} = F_R + \gamma_2 = 1.75\times10^{-2} Nm^{-1}$) must be applied on D2 to keep it so close to D1.

Using Miao et al. calculations[10], Liu et al [3] suggested a quantum-well-action mechanism (QWA) to explain the spontaneous formation of DSF_S in highly doped 4H-SiC during annealing. In our work, the QWA cannot be totally excluded as an extra driving force for the DSF_S nucleation and expansion. Indeed, in the selected temperature range, the Fermi level in our n^+-doped 4H-SiC is

above the lowest energy electron state of the quantum well associated with the DSF_S. Kuhr et al [11] calculated the net energy gain due to electrons entering single or double stacking faults in 4H-SiC as a function of temperature and nitrogen doping concentration through solution of the charge neutrality equation. Using the same method and our temperature and doping concentration conditions, the QWA force applied on DSF_S (F_{QWA}) is found to be about $0.7\times10^{-2}\ N.m^{-1}$ which is of the same order of magnitude but smaller than F_{ADD}. Moreover, in the QWA model Si(g) and C(g) should be produced indifferently. In our work, the C(g) are never observed and are thus assumed to be immobile. These results indicate that the QWA does not play a major role in the DSF_S nucleation and expansion.

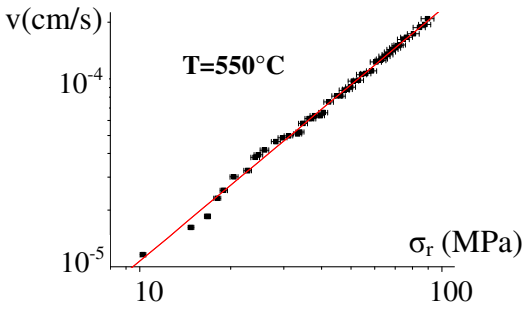

Figure 5 : Variation of the velocity of the 30° Si(g) pairs versus the resolved shear stress at 550°C.

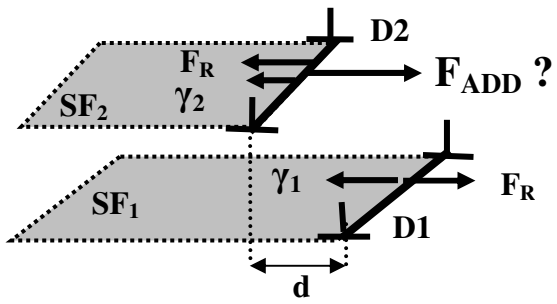

Figure 6 : Scheme of the applied forces on the two identical 30° partial dislocations at equilibrium.

The 4H→3C phase transformation may provide the main extra driving force responsible both of the small equilibrium distance between the two bounding partials and of the high 30° partial mobility. Indeed, the cubic phase is known to be more stable than the 4H one in this temperature range in n-doped SiC and thus the stacking fault energy γ_2 turns out to be negative.

Conclusion

Macroscopic double stacking faults (DSFs) are introduced mechanically in 4H-SiC by cantilever bending and characterized by weak beam and HRTEM. The DSFs are due to the glide of 30° Si(g) partial pairs in adjacent basal planes. The QWA cannot be the unique extra driving force responsible of DSF_S formation and expansion, leaving the 4H→3C phase transformation as the second plausible explanation for the high DSFs mobility.

References

[1] J. Q. Liu, M. Skowronski, C. Hallin, R. Soderholm, H. Lendenmann. Appl. Phys. Lett. 80 (2002). p.749-751

[2] P. O. A. Persson, L. Hultman, H. Jacobson, J. P. Bergman, E. Janzen, J. M. Molina-Aldareguia, W. J. Clegg and T. Tuomi, Appl. Phys. Lett. 80 (2002), p.4852-4854

[3] J. Q. Liu, H. J. Chung, T. Kuhr, Q. Li and M. Skowronski, Appl. Phys. Lett. 80, (2002) p.2111-2113

[4] H. J. Chung, J. Q. Liu, and M. Skowronski, App. Phys. Lett. 81 (2002) p.3759-3761

[5] R. S. Okojie, M. Xhang, P. Pirouz, S. Tumakha, G. Jessen, L. J. Brillson, Appl. Phys. Lett. Vol 79 (2001), p.3056

[6] P. Stadelman, Ultramicroscopy, 21, 131, 1987.

[7] M. Lancin, C. Ragaru and C. Godon. Philosophical Magazine B, Vol. 81, (2001), p.1633-1647

[8] A. Georges, Thèse d'Etat, Nancy-France (1977)

[9] H.P. Iwata, U. Lindefelt, S. Oberg, P.R. Briddon, J. Appl Physics, Vol. 94, (2003), p.4972-4979

[10] M. S. Miao, S. Limpijumnong, R. L. Lambrecht. Appl. Phys. Lett. 79, (2001), p.4360.

[11] T.A. Kuhr, J.Q. Liu, H.J. Chung, M. Skowronski. J. Appl Physics, Vol. 92, (2002), p.5863-5871