Study of Shockley partial dislocation mobility in highly N-doped 4H-SiC by cantilever bending

H. Idrissi[1], **G. Regula**[1], **M. Lancin**[1], **J. Douin**[2], and **B. Pichaud**[*1]

[1] TECSEN, UMR-6122 CNRS, Université Paul Cézanne, Aix-Marseille III, 13397 Marseille-cedex 20, France
[2] LEM, CNRS-ONERA, BP 72, 92332, Chatillon cedex, France

Received 11 September 2004, accepted 9 November 2004
Published online 7 April 2005

PACS 61.72.Ff, 61.72.Lk, 61.72.Nn, 62.20.Fe, 81.05.Hd, 81.05.Je

Well-controlled population of dislocations are introduced in 4H-SiC by bending in cantilever mode and annealing at temperature ranging from 400 °C to 700 °C. The introduced-defects consist of double stacking faults (DSFs) bound by 30° Si(g) partial dislocations. Their expansion is asymmetric with a velocity directly measured on the surface of KOH etched-samples after deformation. Values of the activation energy and the stress exponent are given, and the formation of DSFs is discussed.

1 Introduction

4H-SiC is considered to be very promising for high voltage switching devices. Structural defects such as dislocations and stacking faults have been identified as potentially origin of electrical performance degradation during switching and it is of crucial importance to understand their nucleation and development conditions. The parameters which control dislocation dynamics may be approached indirectly with the help of plasticity experiments and some data are available in the temperature range 500 °C-1400 °C and for n-doped 10^{18}-10^{19}cm^{-3} SiC [1–3]. Up to now, no direct dislocation velocity measurements, either perfect or partial, have been tried which could confirm the evaluation extracted from plasticity works.

In addition during 4H-SiC processing around 1100 °C with similar doping type and doping level, new defects based on double stacking faults (DSFs) associated to partial dislocations have been observed [4–7]. Their origins are still in discussion. One question concerning these defects is: are mechanical stresses developed during the process responsible for their nucleation and/or their development, or are they produced by other mechanisms?

So in order to try to answer this question and to provide direct measurements of the dislocation dynamics parameters, we have nucleated and developed individual partial dislocations under a well-controlled state of stress in 4H-SiC. The defects have been precisely characterised by chemical etching, transmission electron microscopy (TEM) in Weak Beam (WB) and high resolution (HR) imaging modes. The dislocation velocity has been measured as a function of stress and temperature between 400 °C and 700 °C and compared to the parameters extracted from plasticity experiments.

2 Experimental

The N-doping level (5×10^{18} cm^{-3}) of the wafers selected for this study is in the range of earlier plasticity studies on 4H-SiC. The laboratory know-how on dislocation engineering in silicon [8] was adapted to

[*] Corresponding author: e-mail: bernard.pichaud@univ.u-3mrs.fr, Tel.: 334 91 28 83 11, Fax: 33 4 91 2827 93

4H-SiC samples. The latter were cut in (11-20) oriented 4H-SiC N-doped wafers to increase at best the Schmid factor S up to 0.43 in both slip systems [1-210](0001) and [-2110](0001). Thus, a large resolved shear stress σ_r on the (0001) glide plane (drawn in light grey in Fig. 1) can be reached with a low bending amplitude δ, limiting sample breaking. The defects were nucleated by scratching the sample surface in the *X* direction with a diamond tip loaded with a 50g weight. The samples were bent at room temperature around the *Y* direction in cantilever mode, the defect source always under compression.
The resolved shear stress along the sample length $\sigma_r(X,Z)$ was measured by recording the local radius of curvature *R(X)* via the displacement measurement of a laser reflection on the sample surface. Please note that $\sigma_r(X,Z)$ is constant along the sample width *Y* perpendicular to *X*. Thus, σ_r is not constant along the expansion length *D(X)* of the defect since the gliding plane is 45° inclined with the *Y* direction. Moreover, σ_r decreases linearly throughout the depth, changing its sign beyond the neutral plane (see Fig. 1b)

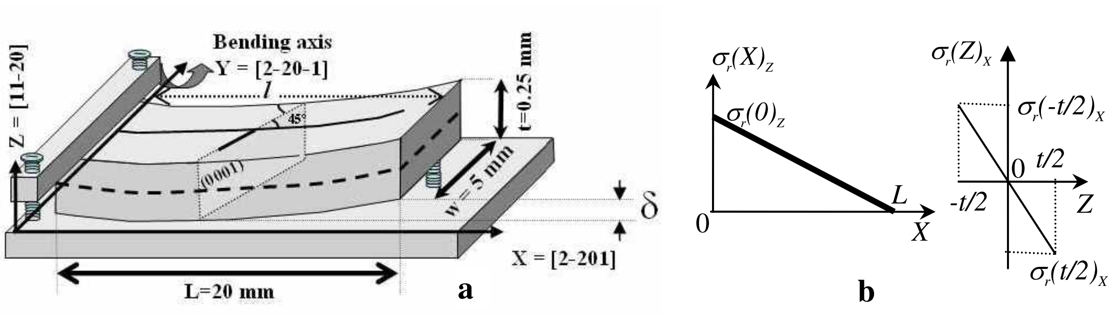

Fig. 1 a) Geometry of the sample on the deformation holder, showing: an (0001) glide plane, an example of the print of the defect path (strait black draw) from the scratch (bent black draw), the neutral plane (dashed curve), the distance *L* between the fixed and the bent sample sides and the flexion amplitude δ. b) Schematics of the variations of $\sigma_r(X,Y)$ for a fixed *Z* along the sample length *L* (bold line) or a fixed *X* throughout the sample thickness *t* .

Then, the samples were annealed under stress in Ar flux at temperature ranging from 400 °C to 700 °C in a conventional furnace. Finally, the defects were characterized by chemical etching with molten KOH (500°C, 10 min), WB and HRTEM techniques performed at 200 kV with the help of Focused Ion Beam (FIB) machining. Below, we will only report the results obtained on the compression side of the sample.

3 Results

3.1 Defect characterization

Figure 2 displays, as an example, optical micrographs of KOH etched-surfaces: the dark straight lines 45° inclined with the scratch are fingerprints of stacking faults (SFs) emerging at the surface. Note that they expand from the scratch in only one direction.

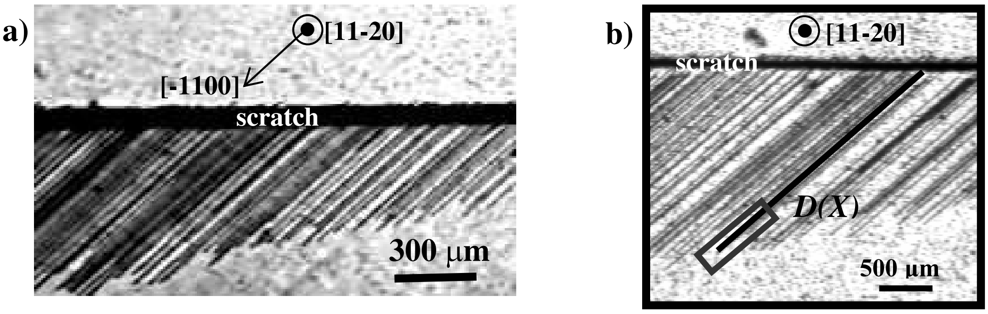

Fig. 2 Optical micrographs after KOH etching. a) and b) Samples deformed at 400 °C, 7.5 h and at 550 °C, 1 h, respectively. In b) the box indicates the location where FIB samples were prepared at a SF tip; *D(X)* is the SF length.

The defect characterization was realized using HRTEM of (11-20) plane views and WB techniques on (0001) sections (Fig. 2b) cut by FIB (focused ion beam) at the end of etched line. An example of the images obtained is shown in Figs. 3a and b. The defect was identified as a double stacking fault (DSF), made of two single faults on successive (0001) planes (Fig. 3a) which corresponds to a 3C-SiC lamellae embedded in 4H-SiC. This DSF was developed by the glide of two identical 30° Shockley partials (Fig. 3b) on two successive glide planes. We have already demonstrated by core reconstruction that they are of Si(g) type [9]. The width of the DSF in the Z direction is close to half the thickness of the sample, so, the DSFs extend from the compression surface up to the neutral plane. Such defects already identified at 550 °C and 620 °C [9] were found identical all over the full 400 °C-700 °C temperature range.
In addition, at the DFS tip, the distance between the two 30° Si(g) is very small (around 60 nm) and does not seem to be strongly influenced by the temperature between 400 °C and 700 °C.

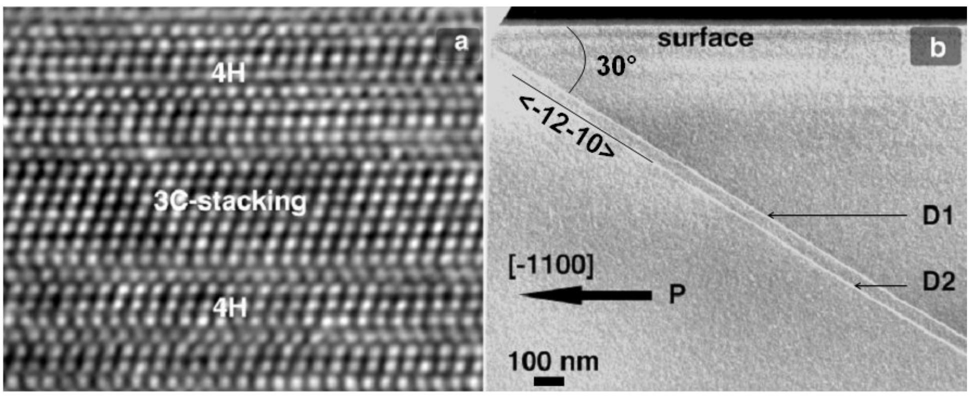

Fig. 3 Micrographs of the DSFs a) [11-20] HR image: sample region double sheared by 30°Si(g) partial dislocations and b) WB image taken near the [0001] zone axis with g = -12-10: the 30°Si(g) partial dislocations D1 and D2 lay along two parallel Peierls valleys in the (0001) glide plane.

Two sets of DSFs were observed. One of them corresponds to the straight black lines in Fig. 2, the length of which is clearly a function of the compression stress due to bending. The other one consists of very short-path DSFs close to the scratch and were not driven by the compression stress. We used only the first set for velocity measurements.

3.2 Mobility of the 30° Si(g)

The mobility v of the partials at the DFS tip is obtained by measuring the length *D(X)* of the etched trace (Fig. 2b) and dividing it by the annealing time, *X* being the coordinate corresponding to the half distance crossed by the partials. The corresponding $\sigma_r(X, Z=t/2)$ is an average of the surface stress underwent by the defects during their motion. Moreover, σ_r decreases along the partial segments when they go deeper and deeper in the sample. It was demonstrated [10] on the basis of the Hirth and Lothe theory for double kink movement [11] that under such varying stress the dislocation moves always parallel to the Peierls valleys and that the representative value of the stress σ_{eff} which must be used to couple with the measured velocity is the mean stress underwent by the dislocation segment, that is the one at its mid point between the surface and the neutral plane i.e. $\sigma_{eff} = \sigma_r(X, Z=t/4)=1/2\ \sigma_r(X, Z=t/2)$. Figure 4 displays the results of our velocity measurements for the 30° Si(g) partial dislocations in the temperature range 400 °C-700 °C. In Fig. 4a one can remark that the velocity follows the classical power law with the stress, the stress exponent being close to 1.8 except for 470 °C. In that particular case, in the lower stress range, the measurements were impacted by some nucleation problems. Figure 4b is an Arrhenius plot of the velocity as a function of temperature drawn for σ_{eff} = 50 MPa. The extracted value of the activation energy for 30° Si(g) Shockley partial dislocation is Q = 1.4 ± 0.2 eV.

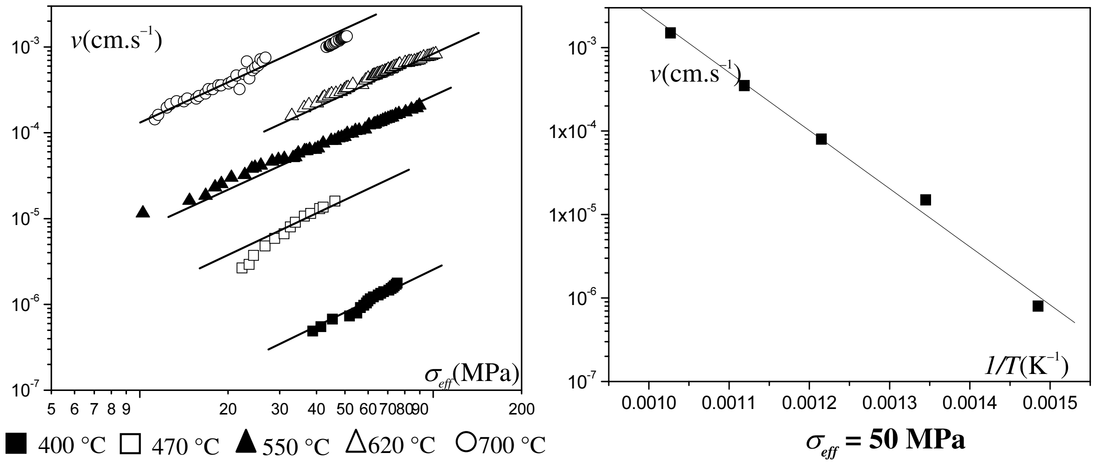

Fig. 4 30° Si(g) partial velocity measurements a) as a function of stress at different temperatures and b) versus the reverse of the temperature.

4 Discussion

Using an external stress, we have nucleated and developed 30°Si(g) Shockley partial dislocations and DSFs and we have studied their dynamics. The main driving force responsible for the DSFs expansion is the applied stress as their expansion is clearly controlled by the compression stress and by the temperature following the classical phenomenological law:

$$v = A\left(\sigma_{eff}\right)^m exp\left(-Q/kT\right) \tag{1}$$

This is in contradiction with some authors affirmations [6] that DSFs are not produced by stress but by electronic effects (quantum well effect, see below) based on the observation of both Si core and C core partials bounding the DSFs. In our case the DSFs are only bounded by 30°Si(g) partial dislocations.

Our results show similarities with those based on plasticity experiments for temperature below 1100 °C i.e in the brittle domain [1-3] and for similar doping type and concentration. We only observed the glide of 30°Si(g) partial stretching faults. We never detected 90°Si(g) partial dislocations probably because their mobility, being larger than the one of 30°Si(g), allows them to leave rapidly the samples. At the very emerging point of the moving dislocations the image forces probably act to rock them normal to the surface in 90° orientation but this 90° Si(g) segment never exists in our WB images. Either they have left the sample or our preparation treatments have removed them. Finally, we did not observe any C(g) dislocations which confirms the previous experimental data.

However, our results also reveal striking points. We observe only DSFs, never SSFs which is quite different as compared to plasticity experiments. This difference may be due to the difference in the deformation process. When compare DSF and SSF for an equivalent shear amount, that is a DSF and two SSFs having the same surface the advantage for the DSF is a gain in stacking fault energy $\gamma_1+\gamma_2$ instead of $2\gamma_1$ for the SSFs, γ_1=1.8 10^{-2}J.m^{-2} [12], γ_2 being the energy of the second SF when the first already exists γ_2=0.3 10^{-2}J.m^{-2} [12]. The disadvantage is the elastic energy corresponding to the interaction of the very close partials bounding the DSF. The relative weight of these contributions depends on the size of the defect (SSF or DSF). For very low defect density and very large defects as in our case, the surface contribution (stacking fault energy) dominates and DSF are more stable, whereas for plasticity experiments the defect density is much larger and the defects smaller favoring the line contribution (interaction energy) that is SSFs. Anyway this indicates that 3C-SiC lamella are very easy to produce and that this quantum-well-like structure is more stable than single stacking faults (SSFs) at least in our temperature range.

Another important result is related to the quite low value of the activation energy for 30°Si(g) Shockley partial glide we got from our experiments. We found 1.4 ± 0.2 eV which is significantly lower than the estimated values extracted from plasticity experiments 1.8 - 2.1 eV [1-3] in the brittle regime. This value is even lower than the activation energies in Si and Ge [13, 14]. Moreover, the velocities measured at the tip of the DSFs for 30°Si(g) partial dislocations are even higher than those of perfect dislocations in silicon in the same temperature and stress ranges. This indicates that additional phenomena are involved in the 30°Si(g) partial mobility in our case. The well known recombination enhanced mobility mechanism cannot be invoked here since it would need ultra-violet light. To understand the origin of additional driving forces which should be considered, one can use the geometry of the two 30°Si(g) partial dislocations bounding the DSFs (Fig. 5). The equilibrium distance between the partials is small, 60 nm as an average, the different forces (per unit length) applied on both partial dislocations are: i) the repulsive force $\boldsymbol{F_R}$ between the two 30° Partial which could be estimated to 1.8 10^{-2} J.m^{-2}, ii) the force γ_1 due to the fault SF1 on dislocation D1, iii) the force γ_2 due to the fault SF2on dislocation D2 (see values above).

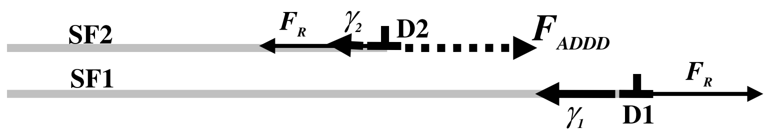

Fig. 5 Scheme of the forces involved in the equilibrium of the 30° Si partials at the tip of the DSF.

To get the equilibrium, an additional force should be added $\boldsymbol{F_{ADD}} = \boldsymbol{F_R} + \gamma_2$ =2.1 10^{-2}J.m^{-2} which can push D2 toward D1 favoring the extension of the second fault until the repulsion between dislocations becomes too high. What is the nature of this force? Is it simply due to a spontaneous 4H- 3C phase transformation which in that case would give a negative value for γ_2 or/and to another phenomenon? For instance it was suggested [15] a quantum well like effect. When the Fermi level is above the energy level of the DSF 0.6-0.7 eV below the conduction band, that is for doping level above 10^{18} cm^{-3}, an energy gain can be obtained which could provide the additional force considered above. An estimation of this contribution, using the model by Kuhr et al.[4] gives ~10^{-2} J.m^{-2} which is not enough but may help the transformation. Obviously all these values are rough estimations (even the stacking fault energies) and additional works are needed to fully understand the DSF and the accompanying Shockley partials dynamics.

References

[1] A. V. Samant, M. H. Hong, and P. Pirouz, phys. stat. sol. (b) **222**, 75 (2000).
[2] J.L. Demenet, M.H. Hong, and P. Pirouz, Mater. Sci. Forum **338-342**, 517 (2000).
[3] J.L. Demenet, M.H. Hong, and P.Pirouz, Scripta Mater. **43**, 865 (2000).
[4] T.A. Kuhr, J.Q. Liu, H. J. Chung, M. Skowronski, and F. Szmulowicz J. Appl. Phys. **92**, 5863 (2002).
[5] J.Q. Liu, H.J, Chung, T. Kuhr, Q. Li, and M. Skowroski, Appl. Phys. Lett. **80**, 2111 (2002).
[6] H.J. Chung, J.Q. Liu, and M. Skowronski, Appl. Phys. Lett. **81**, 3759 (2002).
[7] R.S. Okojie, M. Xang, P. Pirouz, S. Tumakha, G. Jessen, and L.J. Brillson, Appl. Phys. Lett. **79**, 3056 (2001).
[8] J.L. Mariani, B. Pichaud, F. Minari, and S. Martinuzzi, J. Appl. Phys. **71**, 960 (1992).
[9] H. Idrissi, M. Lancin, J. Douin, G. Regula, B. Pichaud, R.El bouaydi, and J.M. Roussel, M.R.S. Conf. Proc. (2004), to be published.
[10] B. Pichaud, P. Jean, and F. Minari, Philos. Mag. A **54**, 479 (1986).
[11] J.P. Hirth and J. Lothe, Theory of dislocations (Malabar, FL:Kreiger, 1992), p. 531.
[12] H.P. Itawa, U. Lindefelt, S. Oberg, and P.R. Brindon, J. Appl. Phys. **94**, 4972 (2003).
[13] A. George and J. Rabier, Rev. Phys. Appl. **22**, 941 (1987).
[14] T. Kruml, D. Caillard, C. Dupas, and J.L. Martin, J. Phys.: Condens. Matter **14**, 12897 (2002).
[15] M.S. Miao, S. Limpijummong, and R.L. Lambrecht, Appl. Phys. Lett. **79**, 4360 (2001).