

## Thermoplastic Deformation and Residual Stress Topography of 4H-SiC Wafers

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### ABSTRACT

We have measured thermoplastic deformation in as-received, *single-side polished*, 4H-SiC wafers and also residual stresses in homoepitaxially grown epilayers on wafers by radius curvature measurements. The wafers studied had n-type resistivities of 0.010-0.011  $\Omega$ -cm and p-type resistivities of 4.42, 4.72, 9.57  $\Omega$ -cm. In a first thermal excursion to 900 °C in vacuum, the bow height of the bare substrates in all cases decreased with temperature. Upon cooling down, however, the bow heights remained largely unchanged from their values at 900 °C. A second cyclic excursion to 900 °C did not yield any significant change in the curvature, thus indicating that the substrates had thermoplastically deformed in the first heating cycle. Epilayers having nitrogen doping between  $5 \times 10^{17}$  and  $2 \times 10^{19}$   $\text{cm}^{-3}$  grown on the n- and p-type substrates resulted in compressive stresses ranging between 190 and 400 MPa in the epilayers. Transmission electron microscopy (TEM) examination of the n-type epilayer (with doping levels of  $5 \times 10^{17}$   $\text{cm}^{-3}$  and  $5 \times 10^{18}$   $\text{cm}^{-3}$ ) on the n-type substrate, revealed bands of stacking faults (SFs) confined within the epilayers after the bicrystals were further annealed at 1150°C in nitrogen for thirty minutes. These doping levels are approximately one and two orders of magnitude below the reported threshold value of  $3 \times 10^{19}$   $\text{cm}^{-3}$  previously suggested for the onset generation of SFs in annealed n-type 4H-SiC epilayers. The calculated residual stresses in all the epilayers were above the critical stress for the motion of dislocations above 1000 °C in 4H-SiC. Thus the SFs that form by glide of pre-existing partial dislocations may actually be stress induced and occur across a much wider range of doping levels. Therefore, it is possible that a significant mechanism for formation of the stacking faults and 3C bands observed in thermally treated 4H-SiC wafer is stress relief via the generation and motion of new and pre-existing partial dislocations on the basal planes of 4H-SiC.

### INTRODUCTION

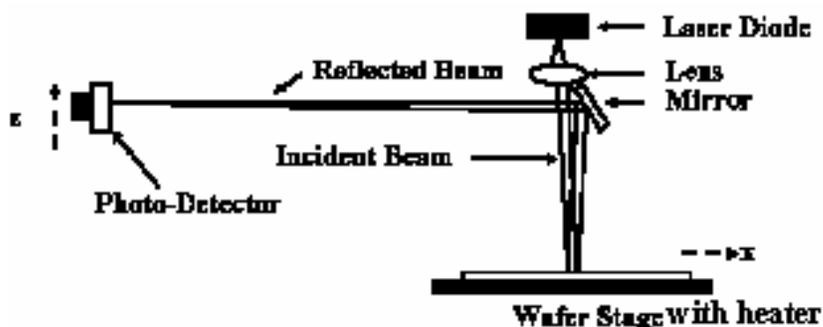
The deleterious effects of internal stresses on the yield and reliability of semiconductor electronic devices continue to remain a major research topic [1]. In silicon carbide (SiC) technology, the production of bulk crystals for high volume commercialization is primarily motivated by the advantages provided by the superior electronic properties of this material (i.e., high breakdown field, wide bandgap, and high thermal conductivity) over traditional semiconductors [2]. These properties will allow SiC-based devices to support high speed and high voltage switching, and to operate in much higher temperature and radiation environments than conventional semiconductor devices. However, the magnitude of stresses that develop during bulk growth of single crystal SiC could be high enough to introduce structural defects such as dislocations (i.e., basal plane dislocations and micropipes) [3,4]. Basal plane dislocations (BPDs) are known to be dissociated into two partial dislocations bounding a ribbon of stacking fault [5]. Under certain conditions of stress and temperature, the partials move and greatly expand the stacking faults (SFs); it is the latter that are thought to be responsible for the degradation observed in SiC PiN diodes during operation [6]. The SFs generated during thermal processing can also degrade device integrity due to the unexpected changes in the electronic properties

of the crystal [7,8]. According to Lindefelt and coworkers, a SF in 4H-SiC acts as a one-dimensional quantum well, thus altering the physical and electronic properties of the crystal [9]. This idea is based on total energy calculations of a 4H-SiC crystal containing an intrinsic SF, where it has been found that a narrow band is split off from the bottom of the conduction band and extends about 0.2 eV into the bandgap of 4H-SiC [10]. The observation of SFs leading to the formation of 3C-like bands in n-type 4H-SiC epilayer doped  $1.7 \times 10^{19} \text{ cm}^{-3}$  after routine oxidation or argon annealing at  $1150^\circ\text{C}$  has been reported previously [7,8,11]. Liu and coworkers proposed the formation mechanism of the SFs under such annealing conditions to be a spontaneous process where thermally generated carriers are trapped in the quantum well thus lowering the energy of the crystal [11]. Consequently, Kuhr *et al.* calculated a threshold nitrogen doping level of  $3 \times 10^{19} \text{ cm}^{-3}$ , below which SF generation by thermal treatment is not expected to occur [12].

The focus of this work was to conduct a direct measurement of the thermal deformations in the bare 4H-SiC wafers in terms of changes in radius of curvature and bow height. The effect of thermal treatment on some of the epilayers was then analyzed with conventional and high resolution (HR)TEM.

## EXPERIMENT

Six wafers (3 n-type and 3 p-type) of bare *single-side polished*, off-axis, Si-face, (0001)-oriented, 2-inch diameter 4H-SiC were commercially procured [13]. Each wafer was placed in the heating chamber of a temperature controlled integrated metrology tool [14]. The tool employs an optical measurement technique whereby a diode laser beam scans across the diameter of



**Fig. 1:** Schematic diagram of the integrated metrology optical measuring tool [14].

the wafer that is placed on the tips of metal tripods. The tripods extend out 2 mm above the plane of the heater plate; this arrangement provides very small contact areas with the wafer. The reflected beam undergoes a secondary reflection by a mirror and is detected by a precision position photo-detector as shown in Fig. 1. By scanning the diameter of a wafer while the reflected beam is continuously detected, the wafer bow along the entire wafer is obtained, thereby allowing for the direct calculation of the spatially distributed radius of curvature  $R_o$ , of the as-received wafer. Several scan paths along the diameter of each wafer were chosen in order to obtain a topographical map of the surface. Subsequently, the wafer was gradually heated to  $900^\circ\text{C}$  in vacuum (approx.  $10^{-6}$  Torr) from 50 up to  $500^\circ\text{C}$  or  $900^\circ\text{C}$  and back down to  $50^\circ\text{C}$ . Prior to the availability of the  $900^\circ\text{C}$  tool, earlier experiment was conducted on the metrology tool that was capable of handling only up to  $500^\circ\text{C}$  [15]. The ramping rate for both the up and down cycles was  $6^\circ\text{C}/\text{min}$ . The wafer was continuously scanned during heat-up and cool-down to determine its instantaneous radius of curvature,  $R_T$ , at any temperature. The final curvature,  $R_{o2}$ , of the wafer was obtained after it cooled down to room temperature. Thus the thermal deformation history of the wafer was acquired *in-situ*. Next,  $2 \mu\text{m}$  thick homo-epilayers with nitrogen doping levels of  $5.2 \times 10^{17}$ ,  $3.9 \times 10^{18}$ , and  $2 \times 10^{19} \text{ cm}^{-3}$  were commercially grown on the three bare p-type substrates. N-type epilayers of similar thickness, but with nitrogen doping levels of  $5 \times 10^{17}$ ,  $5 \times 10^{18}$  and  $2 \times 10^{19} \text{ cm}^{-3}$ , were grown on the bare n-type substrates. Following homoepitaxial growth, another round of radius of curvature measurements, was performed on the epilayer/wafer system to obtain  $R_{epi}$ . The n-type epilayer/n-type substrate batch was annealed in a nitrogen ambient at  $1150^\circ\text{C}$  in a furnace for thirty minutes. Finally, the annealed wafers were diced into  $5 \times 5 \text{ mm}^2$  square foils and

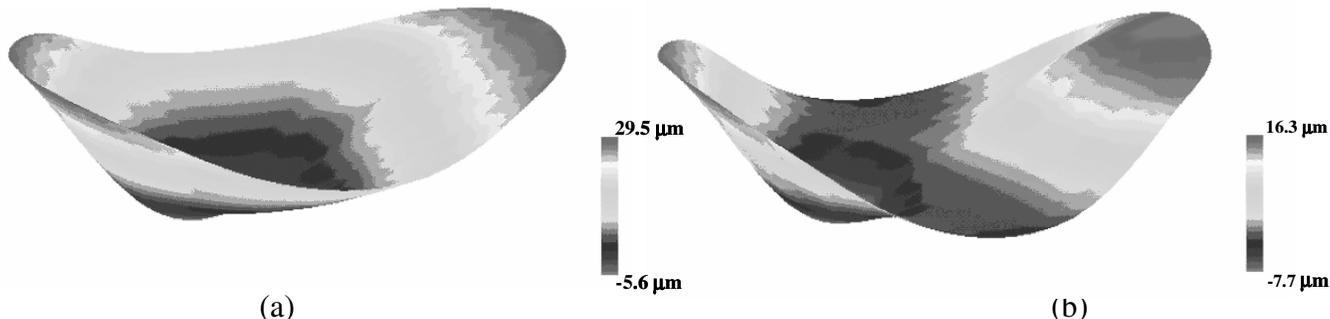
analyzed by TEM. From the radius of curvature after epilayer growth,  $R_{epi}$ , the biaxial stress in the epilayer could be calculated by using Stoney's equation [16]:

$$\sigma_f = E_s d_s^2 [6(1-\nu)Rt_f]^{-1} \quad (1)$$

where  $E_s$ ,  $d_s$ , and  $\nu$  are the substrate Young's modulus (Pa), thickness (m), and Poisson's ratio, respectively;  $t_f$  is the epitaxial film thickness (m), and  $1/R = [1/R_{epi} - 1/R_{cool}]$  ( $m^{-1}$ ).

## RESULTS AND DISCUSSION

All the six as-received bare wafers exhibited various degree of concavity expressed in terms of bow height as shown in the representative data of Figs. 2a and b that depict the physical topography of one of the bare p-type wafer sample P3. It could be seen that the starting material exhibited a warpage that was not necessarily symmetrical (see Fig. 2a). After heating to 900 °C and cooling, the topology of the wafer had changed from its as-received condition with pronounced hysteresis (Fig. 2b). The wafers in all cases became less bowed (increased radius of curvature) after the first thermal excursion. A repeated excursion to 900 °C did not produce any new change in the warpage. Table 1 is the comprehensive results obtained on all the wafers.



**Fig. 2** Representative curvature topography in terms of bow height ( $\mu\text{m}$ ) of a) as-received and b) 900 °C vacuum annealed bare p-type 4H-SiC wafer P3 prior to homoepitaxial n-type epilayer growth.

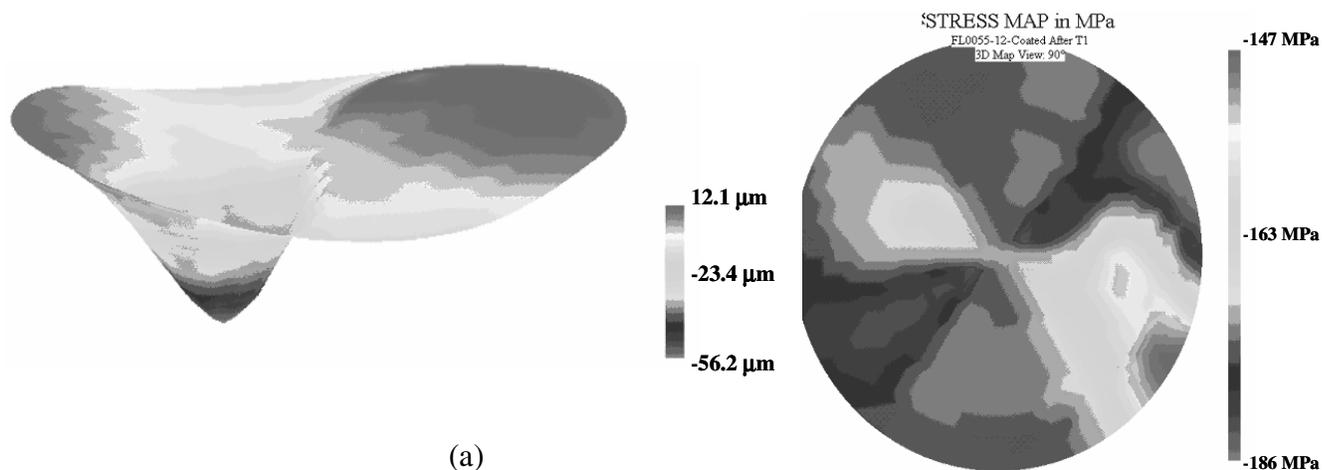
The initial  $R_o$  (in the bare wafer before epilayer growth) was believed to be due to the cumulative residual stress introduced during boule growth and wafer preparation steps (sawing, and lapping). This further confirmed the previously published studies with regard to the thermoplastic characteristics of hexagonal SiC [15,17]. The radius of curvature measured after cooling down from either 500 or 900 °C was found to have increased above the initial room temperature value. It is

Sample	$R_o$ (m)	$R_T$ (m)	$R_{O2}$ (m)	Epi Doping ( $\text{cm}^{-3}$ )	$R_{epi}$ (m)	Epi Stress (MPa)
P1(900 °C)	10.47	28.22	18.63	$2 \times 10^{19}$	35.83	-199.6
P2(900 °C)	8.53	17.16	12.84	$3.9 \times 10^{18}$	19.7	-211.2
P3(900 °C)	10.87	21.66	20.42	$5.2 \times 10^{17}$	41.31	-190.0
N1* (500 °C)	13.2	15.6	15.6	$2 \times 10^{19}$	32.5	-251
N2* (500 °C)	38.6	58.4	76.7	$5 \times 10^{18}$	-24.4	-406.1
N3* (500 °C)	18.6	22.1	23.6	$5 \times 10^{17}$	-122.9	-376.1
4* on-axis	6.9	7.7	6.4	N/A	N/A	N/A

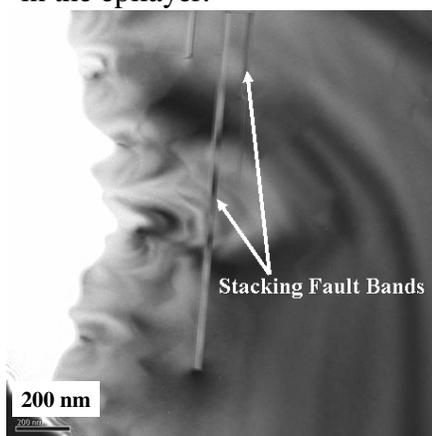
**Table 1:** Changes in wafer radius of curvature before epilayer growth,  $R_o$ , and in the epilayer/wafer bicrystal after growth,  $R_{epi}$ , and the corresponding stress calculated from (1).

\*Wafers annealed up to 500 °C from previously published data [15].

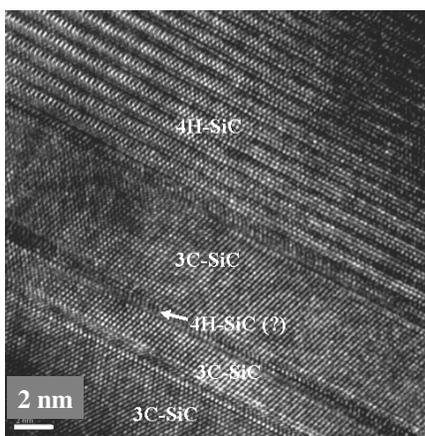
noteworthy that the change in curvature with increasing temperature was more pronounced in the off-axis wafers as compared to the control on-axis wafer (sample 4) in Table 1. From the results shown in Table 1 (includes data from previously published wor[15]), except for sample N2, the radius of curvature of the bare substrates showed only little change on heating to at 500 °C or 900 °C or after cool down. Repeated thermal excursion to 500 °C and 900 °C did not produce any further change in the radius of curvature, indicating that the relaxation was irreversible and that any stress relief occurred via thermoplastic deformation. However, the radius of curvature immediately after epilayer CVD growth,  $R_{epi}$ , showed a dramatic decrease in bow height (increased radius of curvature). Fig. 3a shows the curvature topology of sample P3 in terms of bow height. The resultant topological map of the induced stress in the samples after epilayer growth is shown in Fig. 3b, revealing the non uniform nature of the residual stress in the epilayer. The results shown in Table 1 indicate that the biaxial stresses in the post growth epilayers are compressive with calculated magnitudes between 190 MPa and 410 MPa.



**Fig. 3** a) Curvature topography in terms of bow height ( $\mu\text{m}$ ) of the p-type wafer P3 of Fig. 2 with a homoepitaxially grown  $2\ \mu\text{m}$  n-type epilayer and b) topological map of the corresponding stress (MPa) in the epilayer.

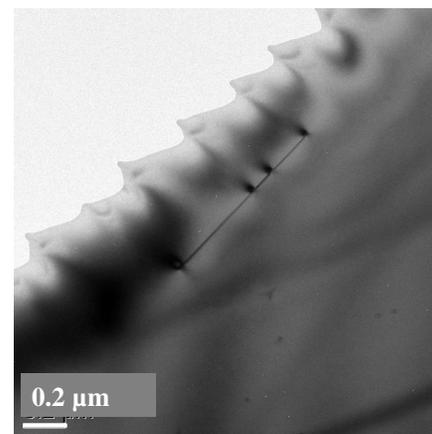


(a)



(b)

**Fig. 4:** Cross sectional TEM image of an off-axis n-type 4H-SiC substrate with a  $5 \times 10^{17}\ \text{cm}^{-3}$ ,  $2\ \mu\text{m}$  epilayer annealed for 30 minutes at  $1150^\circ\text{C}$  in a nitrogen ambient. Note the three SF bands in (a). b) HRTEM of one of the bands showing sub-bands of 4H-SiC that transformed into a 3C-SiC stacking sequence.



**Fig. 5:** TEM image of a SF in the  $2\ \mu\text{m}$  thick 4H-SiC epilayer having a doping level of  $5 \times 10^{18}\ \text{cm}^{-3}$  and grown on an n-type substrate with a doping level of  $1.8 \times 10^{19}\ \text{cm}^{-3}$ .

The TEM examination of the N(1-3) epilayer/substrate bicrystals (with epilayer doping levels of  $5 \times 10^{17}$  and  $5 \times 10^{18} \text{ cm}^{-3}$ ) after thermal excursion to  $500 \text{ }^\circ\text{C}$  in the metrology chamber and further furnace anneal at  $1150 \text{ }^\circ\text{C}$  in nitrogen ambient for thirty minutes revealed SF bands exclusively within the epilayers, as shown in Figs. 4a and 5. Fig. 4b is the HRTEM of one of the bands in Fig. 4a, indicating the existence of multiple SFs that lead to moderately thick layers with a 3C stacking sequence. These doping levels are one and two orders of magnitude below the threshold value proposed in Ref. [12] for the formation of double faults. No SF bands were observed in the  $2 \times 10^{19} \text{ cm}^{-3}$  epilayer, although we had previously observed SFs in epilayer with a doping level of  $1.7 \times 10^{19} \text{ cm}^{-3}$  [7]. The existence of these SFs in annealed 4H-SiC epilayers of such low doping levels cannot be explained with the quantum well action(QWA) model as proposed in Ref. [12] because in these two cases, the Fermi level should lie several tenths of an eV below the  $E_c - 0.2 \text{ eV}$  position of the split-off band [10]. Also significant is the fact that the SF features were not observed in the highly doped ( $3.81 \times 10^{19} \text{ cm}^{-3}$ ) *on-axis* 4H-SiC substrate #4 even after annealing at  $1150^\circ\text{C}$ . This behavior is very different from annealed *off-axis* highly doped 4H-SiC substrate [8]. According to the QWA model, SFs should be generated in thermally treated wafers irrespective of whether they are on- or off-axis. The generation of SFs can be attributed to the expansion of partial dislocations in pre-existing basal plane dislocations (BPDs). By resolving the measured bi-axial stresses in the epilayers of the present work, shear stresses between  $-20$  and  $-70 \text{ MPa}$  along the  $\langle 11\bar{2}0 \rangle$  direction can be obtained. These values are much larger than the experimentally-determined values of critical resolved shear stress for yielding of 4H-SiC at the growth temperature (less than  $1 \text{ MPa}$ ) [5]. It is therefore seen that the shear stress that develops in the epilayer is more than that required to yield the crystal, thus allowing for dissociated dislocations to form and for partials to expand.

## CONCLUSION

This work has shown that single-sided polished 4H-SiC substrates, when subjected to thermal treatment from  $300 \text{ }^\circ\text{C}$  and higher, thermoplastically deform, an effect that is manifested in the warpage of the wafer. Metrology measurements show that compressive residual stresses are introduced in the homoepitaxially grown epilayers on these substrates. Thermal annealing generated SFs in epilayers having doping levels below the previously proposed threshold value of  $3 \times 10^{19} \text{ cm}^{-3}$ , thereby eliminating QWA as a possible driving force at lower doping levels. Considering that the magnitude of the compressive shear stress in the epilayer is much greater than the critical resolved shear stress for yielding of SiC, it is possible that the stacking fault bands observed in many recent thermal annealing experiments are caused by the generation and movement of leading partial dislocations.

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## REFERENCES

- [1] M. Tatsumi, Y. Hosokawa, T. Iwasaki, N. Toyoda, and K. Fujita, *Mat. Sci. and Eng. B* **28**, 65-71 (1994).
- [2] P. G. Neudeck, in *The VLSI Handbook*, edited by W.-K. Chen (CRC Press and IEEE Press, Boca Raton, Florida, 2000), p. 6.1-6.24.
- [3] S. G. Müller, R. C. Glass, H. M. Hobgood, V. F. Tsvetkov, M. Brady, D. Henshall, J. R. Jenny, D. Malta, and C. H. Carter, *J. Cryst. Growth* **211**, 325-332 (2000).

- [4] V. F. Tsvetkov, D. N. Henshall, M. F. Brady, R. C. Glass, and J. C. H. Carter, (*Mater. Res. Soc. Proc.* **512**, Pittsburgh, PA 1998) pp. 89-99.
- [5] M. H. Hong,, A. V. Samant, and P. Pirouz, “Stacking Fault Energy of 6H-SiC and 4H-SiC Single Crystals”, *Phil. Mag. A*, 80(4), 919-935 (2000).
- [6] H. Lendenmann, F. Dahlquist, N. Johansson, J. Bergman, H. Bleichner, and C. Ovren, in *Performance and Reliability of High Power SiC Diodes*, Nara Centennial Hall, Nara Japan, 2000 (Research and Development Association for Future Electron Devices), p. 125-130.
- [7] R. Okojie, M. Xhang, P. Pirouz, S. Tumakha, G. Jessen, and L. Brillson, *Mater. Sci. Forum* **389-393**, 451-454 (2002).
- [8] B. J. Skromme, K. Palle, C. D. Poweleit, L. R. Bryant, W. M. Vetter, M. Dudley, K. Moore, and T. Gehoski, *Mater. Sci. Forum*, **389-393**, p. 455 (2001).
- [9] U. Lindefelt, H. Iwata, S. Öberg, and P. R. Briddon, *Phys. Rev. B* **67**, 155204 (2003).
- [10] M. S. Miao, S. Limpijumnong, and W. R. L. Lambrecht, *Appl. Phys. Lett.* **79**, 4360-4362 (2001).
- [11] J. Q. Liu, H. J. Chung, T. Kuhr, Q. Li, and M. Skowronski, *Appl. Phys. Lett.* **80**, 2111-2113 (2002).
- [12] T. A. Kuhr, J. Liu, H. J. Chung, M. Skowronski, and F. Szmulowic, *J. Appl. Phys.* **92**, 5863-5871 (2002).
- [13] Cree, in *4600 Silicon Drive*, 27703 ed. (Durham, NC).
- [14] Frontier Semiconductor, Inc, 1631 North First Street, San Jose, CA 95112 USA. ([www.frontiersemi.com](http://www.frontiersemi.com)).
- [15] R. S. Okojie, M. Xhang, and P. Pirouz, to be published in the Proceedings of the International Conference on Silicon Carbide and Related Materials, October 5-10, 2003, Lyon, France.
- [16] M. Ohring, *The Materials Science of Thin Films*, Academic Press, Inc., New York. (1992).
- [17] A. Ellison, H. Radamson, M. Tuominen, S. Milita, C. Hallin, A. Henry, O. Kordina, T. Tuomi, R. Yakimova, R. Madar, and E. Janzen, *Diamond and Related Materials* **6**, 1369-1373 (1997).
- [18] D. Hull and D. J. Bacon, *Introduction to Dislocations*, Fourth ed. (Butterworth-Heinemann, Woburn, 2001).