Dislocation Content of Micropipes in SiC

J. Heindl,* W. Dorsch, and H. P. Strunk
Institut für Werkstoffwissenschaften-Mikrocharakterisierung, Universität Erlangen-Nürnberg, Cauerstraße 6, 91058 Erlangen, Germany

St. G. Müller, R. Eckstein, D. Hofmann, and A. Winnacker
Institut für Werkstoffwissenschaften-Werkstoffe der Elektrotechnik, Universität Erlangen-Nürnberg, Martensstraße 7, 91058 Erlangen, Germany
(Received 21 July 1997)

Silicon carbide, a potentially powerful device material, suffers from microscopic hollow defects called micropipes. Their nature is not satisfactorily clarified yet. Our analysis shows that they are hollow core dislocations according to Frank's model, but contain dislocations of mixed type. [S0031-9007(97)05011-4]

PACS numbers: 61.72.Bb, 61.16.Ch, 61.72.Ff, 61.72.Lk

Micropipes are hollow tubes penetrating SiC single crystals along their growth direction ([1], for review see, e.g., [2]) and occur very frequently in SiC. They can be interpreted in the framework of Frank's model of a hollow core dislocation [3,4]: When the magnitude of the Burgers vector of a dislocation exceeds a critical value (approximately 1 nm) it is energetically more favorable to remove the highly strained material around the dislocation line and to create an additional free surface in the shape of a tube. The relation between the equilibrium radius r_0 and the length of the Burgers vector B in the micropipe is given by Frank's formula,

$$\frac{r_0}{B^2} = \frac{\mu}{8\pi^2\gamma}\,. \tag{1}$$

μ: shear modulus (for SiC: $\mu = 1.9 \times 10^{11}$ J/m^3); γ: surface energy of the inner surface of the micropipe. We will use this surface energy as a fit parameter in the following discussion.

We have to note here that Frank's model is applicable to any type of dislocation. However, in the past, because of the easy accessibility, only screw components were considered [4–6] and the obtained results were consequently discussed in terms of "screw dislocations." In the following discussion we will first adopt this notion and present our results in this familiar picture, but later we must modify it by considering an additional edge component of the Burgers vector. In this case, the shear modulus μ in Eq. (1) has to be replaced by the appropriate energy factor K we take for Poisson's ratio $\upsilon = 0.16$ [7].

In former papers [4,5] we investigated by atomic force microscopy (AFM) growth spirals with micropipes in their centers on the as-grown surface of 6H-SiC crystals grown by the modified Lely method, which is a near-equilibrium growth method because of its low supersaturation and growth velocity [8]. We could relate the micropipe radius and the screw Burgers vector component associated with the micropipe. This component can easily be found by adding up the total step height during one revolution of the AFM tip around the micropipe. Upward and downward steps are considered to have opposite signs, and the absolute value of the sum represents the screw component of the micropipe. In this paper we complement those data by adding further measurements of this type. In addition, we consider data recently published by Dudley *et al.* [6]. This group measured by using synchrotron radiation the Burgers vectors (as far as we judge the screw component only) and, by using scanning electron microscopy, the radii of micropipes of commercially available material.

Figure 1 shows all data suitably plotted for a comparison to Frank's formula. There is no unique linear relation between the radius and the square of the Burgers vector as

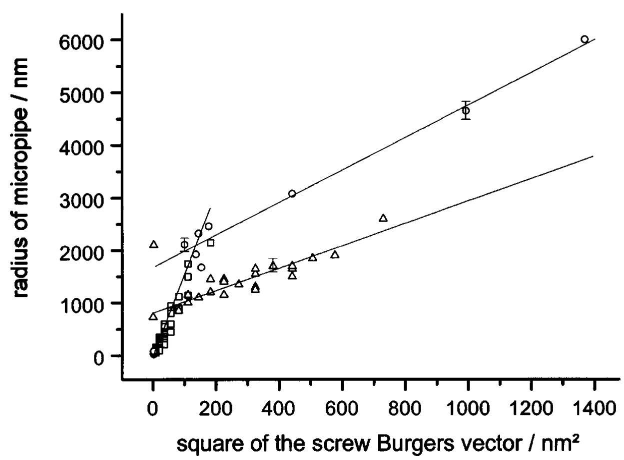

FIG. 1. Screw type Burgers vectors versus radii of micropipes in 6H-SiC. ○: Our published data [5] obtained by AFM. Δ: Our further data obtained by AFM as described in [5]. □: Data from Dudley *et al.* [6] obtained using synchrotron white beam x-ray topography. Three error bars are introduced to indicate the typical errors of the measurements. The fit curves are obtained by least squares fits of subsets of the measured values. It can clearly be distinguished between curves of two different slopes. However, both can be fitted using a surface energy of 0.94 ± 0.08 J/m^2 (see text).

 0031-9007/98/80(4)/740(2)$15.00

required by Eq. (1). We apply least squares fits to three subsets of the data (plotted in Fig. 1). It is appropriate in the present context to distinguish two types of micropipes: type I is represented by the data subset which has a large slope and intersects the origin. Both other data subsets with a small slope represent type II micropipes.

For type I micropipes the evaluation of the slope of the fit curve with the aid of Eq. (1) leads to a physically unreasonable surface energy of 0.18 ± 0.02 J/m^2. This problem can be relieved when assuming that instead of the measured screw Burgers vector a mixed type Burgers vector is associated with the micropipe. In fact, our recent transmission electron microscope work [2,9] has indicated multiples m of (partial) dislocations with mixed Burgers vector $\frac{1}{6}[2, -1, 1]$ (or crystallographically equivalent vectors). (To avoid problems in indexing we use the cubic notation with $[1, 1, 1]$ parallel to the c axis.) This total Burgers vector $m/6[2, -1, 1]$ in the micropipe is lying obliquely to the growth surface, and by measuring spiral steps only its surface-normal (screw) component can be evaluated. The total Burgers vector thus is larger than the observed screw component. From geometrical considerations we obtain the following relation between the magnitude of the total Burgers vector forming the micropipe and its screw component:

$$|B_{\mathrm{total}}| : |B_{\odot}| = 2.13 : 1 . \qquad (2)$$

If we ascribe this mixed Burgers vector now to the type I micropipes, the measured screw component has to be modified for the additional edge component. The dependence of the surface energy on the Burgers vector squared [Eq. (1)] leads to a correction factor of 2.13^2 (due to geometry) and an additional 15% due to the energy factor. This yields a reasonable surface energy of 0.93 ± 0.08 J/m^2.

Large angle convergent beam electron diffraction, a well established method to determine the total Burgers vector of a dislocation was recently applied to nanopipes in GaN [10]. However, thus far we could not successfully use this method because the micropipes in SiC generally have diameters orders of magnitude larger than those of nanopipes in GaN.

We now consider the data subset of the type II micropipes. This set is characterized by a small slope (we take an average value) and by two different offset radii. Straightforward application of Eq. (1) to the average slope yields a surface energy of 0.94 ± 0.04 J/m^2 which is in excellent agreement with the value obtained before. Therefore we deduce that the slopes of the type II micropipe data sets are, in fact, determined by a screw Burgers vector component in the micropipe only (as inferred in the plot of Fig. 1). Then, in consequence, each intersection of these two interpolation lines with the line of the type I micropipe data set indicates a corresponding additional edge Burgers vector component. Thus, according to this analysis, type I micropipes are characterized by a constant ratio of screw and edge Burgers vector components. Type II micropipes are characterized by a constant (more realistically by an almost constant) edge component, and only the screw component varies. The origin of this constant edge component is not known so far. Further work regarding the growth conditions is under way.

There might be a source of a systematic error in the determined micropipe radii due to a possible funnel shape of the micropipe's opening at the surface [3,5]. Thus our measured radii could be somewhat larger than those present in the bulk. This quantitative uncertainty makes the evaluated surface energy a lower limit, but does not affect our conclusions on the nature of the dislocations involved in micropipe formation. In summary, the dependence of the micropipe radius on the Burgers vector content of the observed micropipes in different SiC ingots (ours and other authors') can consistently be explained within Frank's model of a hollow core dislocation when a mixed dislocation is assumed.

This work was supported by the Deutsche Forschungsgemeinschaft (SFB 292 B3) and the Bayerische Forschungsstiftung.

*Electronic address: heindl@ww.uni-erlangen.de

[1] J. Heindl and H. P. Strunk, Phys. Status Solidi (b) **193**, K1 (1996).
[2] J. Heindl, H. P. Strunk, V. Heydemann, and G. Pensl, Phys. Status Solidi (a) **162**, 251 (1997).
[3] F. C. Frank, Acta Crystallogr. **4**, 497 (1951).
[4] J. Heindl, W. Dorsch, R. Eckstein, D. Hofmann, T. Marek, St. G. Müller, H. P. Strunk, and A. Winnacker, in *Proceedings of the 1st European Conference on Silicon Carbide and Related Materials 1996* [Diam. Relat. Mater. **6**, 1269 (1997)].
[5] J. Heindl, W. Dorsch, R. Eckstein, D. Hofmann, T. Marek, St. G. Müller, H. P. Strunk, and A. Winnaker, J. Cryst. Growth **179**, 510 (1997).
[6] M. Dudley, W. Si, S. Wang, C. Carter, Jr., R. Glass, and V. Tsvetkov, in Proceedings of the 3rd European Symposium on X-ray Topography and High Resolution Diffraction, 1996 [Nuovo Cimento D (to be published)].
[7] I. Parfenova, Yu. M. Tairov, and V. F. Tsvetkov, Sov. Phys. Semicond. **24**, 158 (1990).
[8] D. Hofmann, M. Heinze, A. Winnacker, F. Durst, L. Kadinski, P. Kaufmann, Y. Makarov, and M. Schäfer, J. Cryst. Growth **146**, 214 (1995).
[9] J. Heindl, V. D. Heydemann, G. Pensl, and H. P. Strunk, in Proceedings of the 7th Conference on Defect Recognition and Image Processing, Templin, 1997 (to be published).
[10] D. Cherns, W. T. Young, J. W. Steeds, F. A. Ponce, and S. Nakamura, J. Cryst. Growth **178**, 201 (1997).