THE RELATIONSHIP BETWEEN MICROPIPES AND SCREW DISLOCATIONS IN PVT GROWN 6H-SiC

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ABSTRACT

The growth surface of a 6H-SiC boule, grown by physical vapor transport, was examined using scanning force microscopy. The dimensions of surface/micropipe intersections and screw dislocation Burgers vectors have been determined from topographic data. All micropipes are positioned along the lines of super screw dislocations with a Burgers vectors of at least 4 times the c-axis repeat distance (15.2 Å). Perfect c-axis screw dislocations with Burgers vectors of only 15.2 Å are stable and do not have open cores. Measurements show that micropipe core radii, determined indirectly from the width of the craters formed at the surface/micropipe intersections, increase with the square of the dislocation Burgers vector.

INTRODUCTION

Micropipes are cylindrical voids with diameters in the 1-10 μm range that are found oriented along the c-axis of SiC crystals grown from the vapor phase. For example, in crystals grown by the physical vapor transport (PVT) process, these defects occur with a density of 50-10^3 cm^{-2} and extend throughout most of the boule volume [1,2]. Because micropipes limit the operating range of high voltage SiC devices, their elimination has important practical implications [3]. The objective of this paper is to specify the nature of this extended defect.

A possible mechanism for the stabilization of such a long, narrow cylindrical void was proposed by Frank in 1951 [4]. If a dislocation with a sufficiently large Burgers vector threads the length of the crystal, it can be energetically favorable to replace the most highly strained part of the crystal in the vicinity of the dislocation line with an empty cylinder. Frank demonstrated that a state of local equilibrium can be achieved by balancing the elastic energy of the dislocation against the surface energy of facets bounding a narrow cylinder. One of the central predictions of Frank's theory is that the pipe radius (r_0) should be proportional to the square of the Burgers vector (b); the relevant physical parameters that determine the proportionality are the surface energy (γ) and the shear modulus (G):

$$\frac{b^2}{r_0} = \frac{8\pi^2\gamma}{G} \qquad (1)$$

There are several pieces of evidence supporting the idea that micropipes are, indeed, empty core screw dislocations. First, Verma [5] noted that micropipes intersect the growth surface at the origins of optically visible spiral steps. Also, measurements based on optical micrographs of the growth surface have been used to show that the core radius increases with

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the height of the spiral step [6]. More recently, white beam synchrotron topography was used to show that micropipes in PVT grown 6H-SiC crystals were empty core screw dislocations with Burgers vectors 3 to 7 times the c lattice constant (15.2 Å) [7]. In the present paper, we present quantitative scanning force microscopy (SFM) measurements of the dislocation Burgers vectors and micropipe radii based on topographic images of the growth surface of a 6H SiC crystal produced by the PVT method. Our results show that single, perfect c-axis oriented screw dislocations with Burgers vectors of 15.2 Å = b_0 are stable and do not have open cores. On the other hand, all micropipes are found to lie along the lines of super-dislocations with Burgers vectors of nb_0, where n is an integer greater than or equal to 4.

EXPERIMENTAL

All measurements described in this paper were conducted on the same 6H-SiC growth surface. A wafer containing the growth surface was removed from the top of a large single crystal boule grown by the PVT process and imaged without further treatment [1]. SFM analysis was carried out using a Park Scientific Instruments (PSI) scanning probe microscope. Because the scanner field of view was 5 microns, only surface/micropipe intersections smaller than this size were measured. An optical microscope was used to identify the points where the micropipes emerged on the surface and to position the scanning probe. While measurements of more than 25 spirals were made in the course of this study, we compare only a subset of these in the current paper. This subset of measurements was made using exactly the same probe tip (a high aspect ratio PSI ultralever) and instrumental parameters (stage tilt, scan rate, and feedback parameters). While the remaining measurements are consistent with those presented here, limiting our analysis to only this subset allows us to conclude that scatter in the results is not due to differences in the details of the probe shape or the feedback response.

When a dislocation line intersects a surface, the component of the Burgers vector normal to the surface is equal to the height of the step created by the intersection. In this case, we take the screw component of the dislocation's Burgers vector to be equal to the height of the step that emerges from the micropipe. Note that only the component of the Burgers vector normal to (0001) is determined; topographic measurements can not be used to distinguish pure screw dislocations from those of mixed character. Figure 1a shows a typical topographic profile from which a step height was determined. This is a single scan line taken from the image in Fig. 2b, along the fast-scan direction. In order for rotating spiral steps to produce the 6H polytype, they must occur in integer multiples of the c-axis repeat distance, which is the length one perfect screw dislocation along [0001], b_0=15.2 Å. In fact, measured step heights seldom deviated from these intervals by more than a few angstroms (a few percent of the total height). For the final analysis, step heights were assigned to the closest integer multiple of b_0. For example, the height of the step shown in Fig. 1a (136 Å) is very near $9b_0$.

One difficulty associated with determining the core radius from any microscopic examination of the growth surface is that the micropipe radius at the surface is much larger than the radius in the bulk. Because of capillarity, the surface/micropipe intersection forms a sloping crater which is widest at the surface and slowly tapers to the stable radius in the bulk, r_0. A typical SFM line profile extracted from an image of a surface/micropipe intersection is shown in Fig. 1b. The flat part in the center of the depression is where the probe has extended as far as possible into the void (the maximum vertical depth of field is limited by the piezoelectric scanner design and the high voltage amplifiers used in the microscope). Clearly, it is not possible to directly observe r_0 using surface images. However, in Frank's original theoretical description of the empty-core dislocation, he predicted this effect and determined the relationship between the initial slope of the crater, which is determined accurately by SFM, and the ultimate radius [4]. For example, at the point where the slope of the crater wall (dz/dr) is equal to 1/4, the crater radius is 10.5 times r_0. In each case, the crater diameter (d_c) was determined using the points where the slope equaled 1/4 and this value was used to calculate r_0, as illustrated in Fig. 1b.

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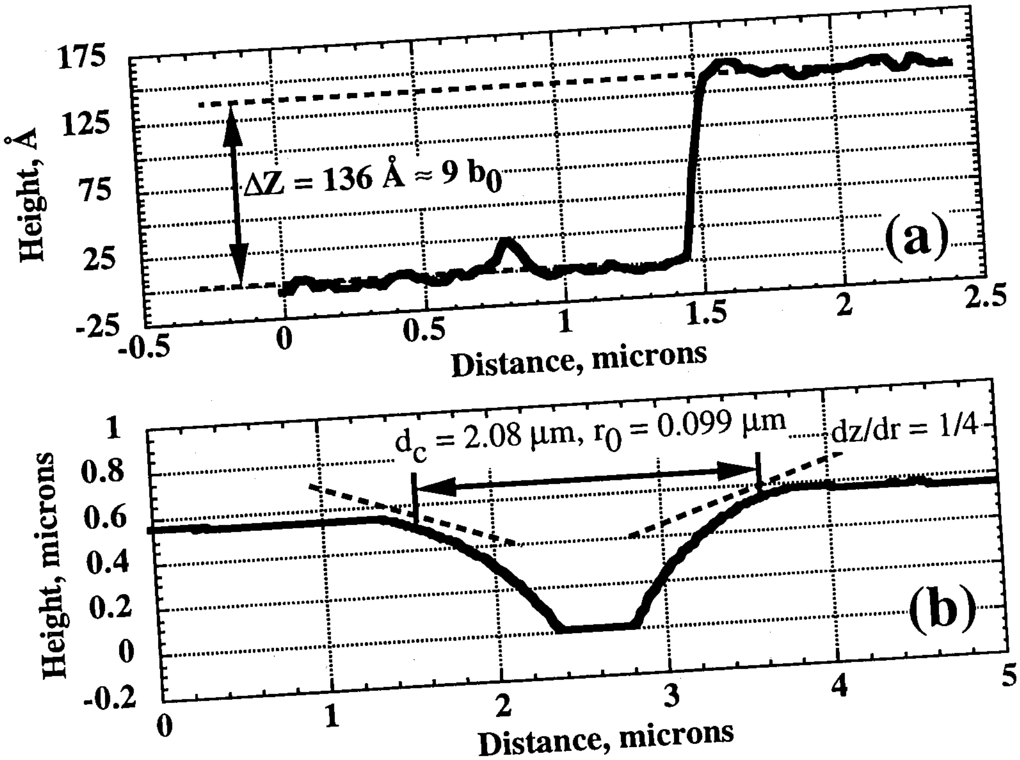

Figure 1. Topographic profiles used to measure the sizes of (a) the Burgers vectors associated with micropipes, b, and (b) the micropipe core radius, r_0.

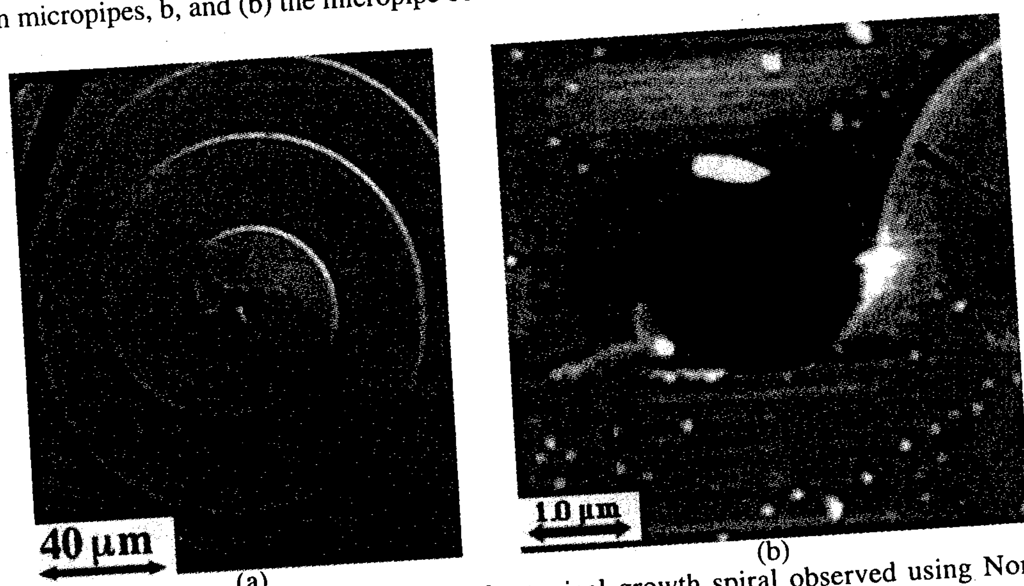

Figure 2. (a) An optical micrograph of a typical growth spiral observed using Nomarski contrast. The small black spot at the end of the spiral step is a surface/micropipe intersection. (b) A SFM image of a different surface/micropipe intersection. The micropipe is the circular black spot in the center, labeled M. Due to the small field of view, only a small portion of the spiral step (labeled S) is shown. A profile taken over this step is shown in Fig. 1a.

RESULTS

The SiC (0001) growth surface was comprised of flat terraces, separated by large, circular growth steps. The flat terraces were decorated with growth spirals such as the one shown in Fig. 2a. A typical SFM image, recorded in the center of a similar spiral, is shown in Fig. 2b. This topographic image is shaded so that the lowest regions appear dark and the highest ones are light. Thus, the roughly circular black spot in the center of the micrograph (labeled M) is the intersection of the micropipe with the surface and the abrupt change in contrast in the upper right hand corner of the image is a step (labeled S) emerging from the pipe. The regions of roughly constant contrast are atomically flat terraces and the white spots are small, ubiquitous, island-like inhomogeneities. A Burger circuit taken around the pipe shows that the height of this step must equal the Burgers vector of a dislocation whose line emerges from the crystal at this point. In this case, the step height (see the profile in Fig. 1a) is 136 Å, so the Burgers vector of this super screw dislocation has a magnitude 9 times that of a single perfect dislocation. In other cases, multiple spiral steps emerged from the same pipe and the sum of the heights was used to determine the total Burgers vector. When the steps are less than 45 Å, or equal to only one or two times b_0, they are not optically visible. In some cases, micropipes were located in the optical microscope without visible spiral steps. However, upon closer examination in the SFM, multiple spiral steps, all with heights of approximately 15 Å, were found to emerge from the defect. Every micropipe examined as part of this study occurred at the point where a super-dislocation emerged from the crystal.

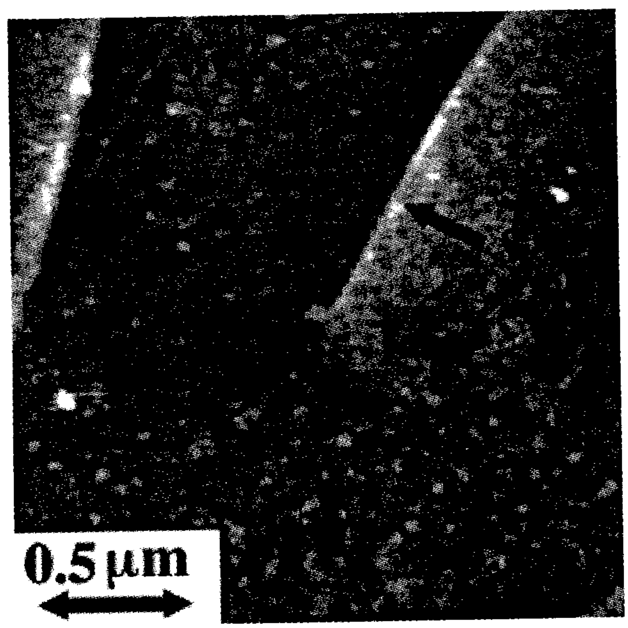

Figure 3. SFM image of a 15 Å step (labeled S) that ends abruptly on a flat terrace without a depression or micropipe.

In some cases, 15 Å spiral steps were observed that terminated on a terrace without a micropipe. One such example is shown in Fig. 3, where a step (labeled S) terminates near the center of the micrograph without an observable depression. Based on studies of surface/dislocation intersections on the surfaces of GaN films using the same instrumentation, we know that it is possible to detect depressions as small as 200 Å in diameter (this is similar to the probe diameter) [8-9]. If there is an even smaller, undetected crater at the end of the step in Fig. 3, the pipe radius associated with such a crater would be less than 10 Å, a physically unrealistic value. Therefore, we conclude that single perfect dislocations with Burgers vectors of 15 Å do not form micropipes.

Based on eqn 1, the micropipe radius should increase with the square of the Burgers vector. Figure 4 shows a plot of b^2 v. r_0 for 9 carefully measured micropipes. Assuming that the shear modulus of SiC is 200 GPa [10] and the surface energy is 4 J/m^2 [11], the slope of this line should be equal to approximately 16 Å; the dashed line on the graph shows this "ideal" slope. Although there is a considerable amount of scatter, the results are consistent with the predicted slope.

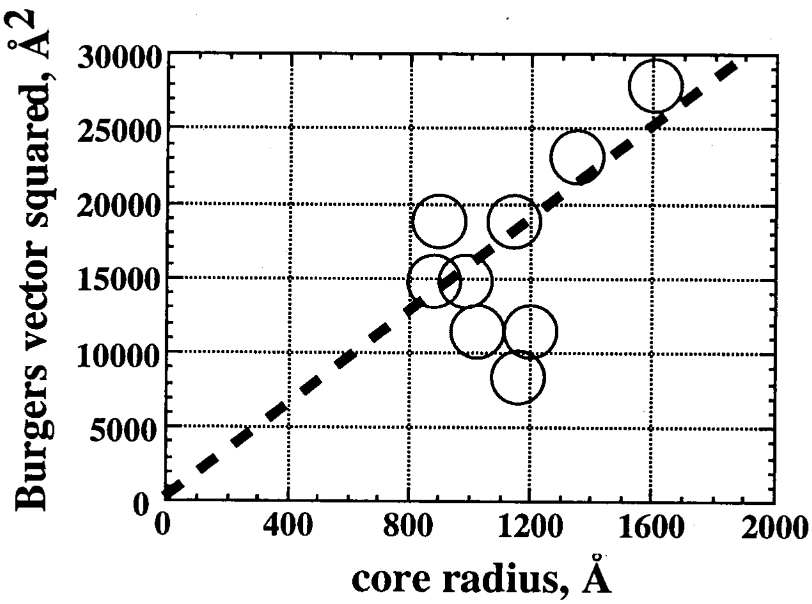

Figure 4. The core radius v. the square of the Burgers vector for 9 carefully measured micropipes. The size of the circle is used to indicate the maximum estimated uncertainties in the determination of b and r_0. The dashed line has a slope of 16 Å.

DISCUSSION

Based on our observations, Frank's theory for the open core screw dislocation seems to provide an appropriate description of the micropipe defect in SiC. The smallest possible perfect dislocations, with Burgers vectors of only 15.2 Å, have stable cores and do not form micropipes. The larger super-dislocations, however, which have more elastic strain, do form the cylindrical voids and the radii of these defects are correlated to the square of the dislocation Burgers vector.

It is, however, important to note that the observed deviations from the predicted behavior are larger than the estimated uncertainties involved in the measurement. We can identify two possible sources for this error. First, Frank's analysis predicts the dimensions of equilibrium defects [4]. The structures described here were formed on a growing crystal. In this case, the core radii should be influenced by the supersaturation of the vapor above the growing crystal surface. In particular, the kinetic radii should be systematically smaller than the predicted equilibrium radii and this contraction can be computed with knowledge of the critical radius for two dimensional nucleation, r_c [12]. We can use the observed spiral terrace width to estimate that r_c is on the order of 1000 Å [13] and, based on this estimate, use the results in ref. 12 to find that the kinetic core radius is about 10% less than the equilibrium core radius. Since this is comparable to other experimental uncertainties, we conclude that kinet effects can not account for the observed deviations.

A second possible cause for the deviations is that the shapes of the craters a influenced by second phase inhomogeneities that are frequently found on the growth surfa and always found on the inner wall of the surface crater at the micropipe. For example, in Fi 2b, a white elongated feature is seen near the top of the micropipe. Similar features are fou near all micropipes and because some of them have influenced the shape of growing steps, w can be certain that they were present on the surface during growth. Such inhomogeneiti might pin the walls of the craters and make them artificially large, an effect that would accou for deviations below the ideal line on Fig. 4. The role that these inhomogeneities play i micropipe nucleation will be described in a forthcoming paper.

CONCLUSION

Scanning force microscopy measurements have been used to demonstrate that micropipes for in SiC to relieve strain at the cores of dislocations with large screw dislocation component Single complete screw dislocations are stable and micropipes have been observed to occur onl when the dislocation Burgers vector is at least 4 times that of the single dislocation. Preventin the formation of these super-dislocations should suppress micropipe formation in SiC.

ACKNOWLEDGMENTS

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