Correlated reduction in micropipe cross sections in SiC growth

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We reveal a correlated reduction in the cross sections of two neighboring micropipes (MPs) in the crystal growth of silicon carbide using computer simulation of phase contrast images. The correlated reduction is explained by the exchange of full-core dislocations in a contact-free reaction between two parallel MPs. We develop a theoretical model that describes the energetics of this process. © *2008 American Institute of Physics*. [DOI: 10.1063/1.2998572]

Nowadays research pertaining to silicon carbide (SiC) power electronics has been hampered by insufficient quality and size of SiC ingots. Seeded sublimation growth techniques produce large-area SiC wafers; however, the sublimation growth is accompanied by the formation of cylindrical pores called micropipes (MPs). As a rule, they represent the hollow cores of superscrew dislocations with giant Burgers vectors. MPs have deleterious effect on the properties of SiC such as large breakdown electric field strength, high thermal conductivity, etc. Therefore, the problem of growing bulk SiC crystals with low MP density is of special importance.[1–5] The MP evolution during the crystal growth is a complex process that involves the formation of new MPs,[6–17] the overgrowth of the existing MPs due to their various reactions with one another,[18,19] and the coalescence of MPs resulting in micropore formation.[3,15,20,21]

In this letter we discuss one more form of MP evolution, namely, a correlated reduction in the cross sections of two neighboring MPs along their axes (generally parallel to the growth direction). Such variations in MP cross sections can lead to MP healing. We provide experimental evidence of this effect and discuss its reasons and a possible mechanism. Our main idea is that MPs can remotely interact with each other by the exchange of full-core dislocations. We propose a theoretical model describing the energetics of this process.

SiC sample was an axial-cut slice along the growth direction [0001] obtained from 4*H*-SiC boule. The boule was grown in Ar by the sublimation sandwich method[9] at the growth temperature of 2100 °C and with the growth rate of 0.5 mm h^{-1}. The sample contained MPs of $\approx$0.5–10 μm in diameter located almost parallel to the surface. MPs were examined with the white beam phase contrast imaging method[3,14,18,20] at the 7B2 X-ray Microscopy Beamline of the Pohang Light Source, Korea. The source with effective sizes of 160 (H) and 60(V) μm was located at a distance of 34 m from the sample. The sample (rotated to have a horizontal position of the MP axis for better edge enhancement in a more coherent vertical direction) was sequentially placed at different distances from the detector. We obtained nine images for the distances from 5 to 45 cm starting with the first image registered at 5 cm from the sample. Three MPs forming a group were examined: two in contact and the third lying remotely. The phase contrast image of the MP group is shown in Fig. 1(a), while Fig. 1(b) demonstrates the optical micrograph of the remote MP, taken on a Zeiss universal microscope equipped with a charge coupled device.

For all sample-to-detector distances and for different points along the axis of MPs, the intensity profiles normal to the axis were measured. To determine the characteristic sizes of the MP cross sections in different points along the MP axis, we applied the method of computer simulation[22] of the measured intensity profiles with the special assumption that these sections can have variations in longitudinal (along the beam) and transverse (across the beam) sizes. For every MP cross section under investigation, the computer program calculated many profiles for various possible section sizes on the base of Kirchhoff propagation[22] to find the profile, which gives the best fit to the individual experimental profile registered for this section. The coincidence allowed us to determine both the longitudinal and transverse sizes of the section (Fig. 2).

The computer simulation showed that the transverse sizes of the MP sections are several times larger than their longitudinal sizes, indicating elliptical cross section. The reasons of such nonroundness are not clear yet. As one possibility, MPs can have elliptic cross sections if their dislocation Burgers vectors have not only a screw, but also an edge component. The other possibility is in the origin of these MPs. If they are formed from the slits at the boundary of a foreign polytype inclusion, they simply could not change yet their shape during the crystal growth.

We note that the transverse sizes drastically change during the crystal growth in contrast to the longitudinal sizes, which remain approximately the same (and of the order of

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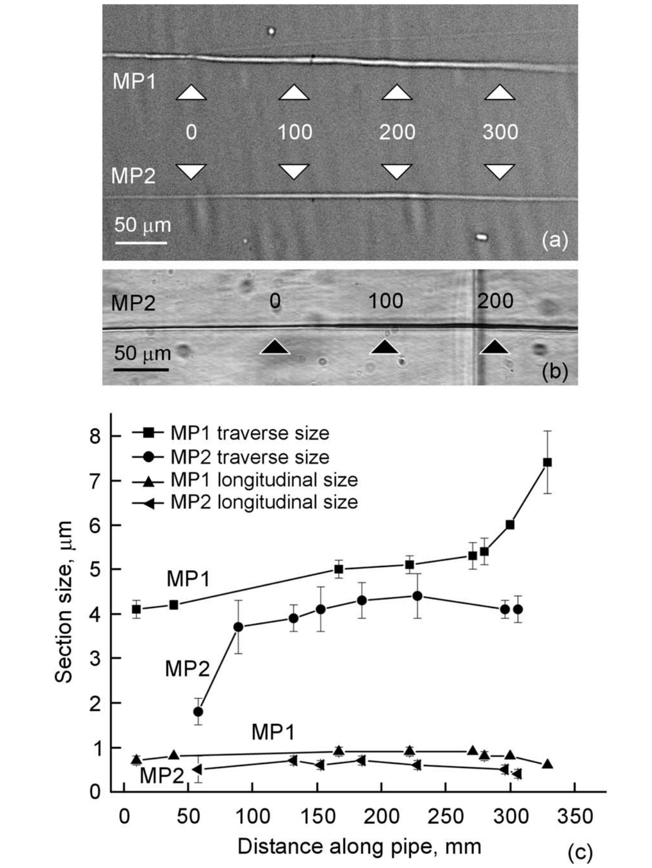

FIG. 1. (a) Phase contrast image of MP group; (b) optical micrograph in transmission light of MP2 lying remotely from two MPs in contact; (c) variation in cross-section sizes along the axes of MP1 and MP2 from the group.

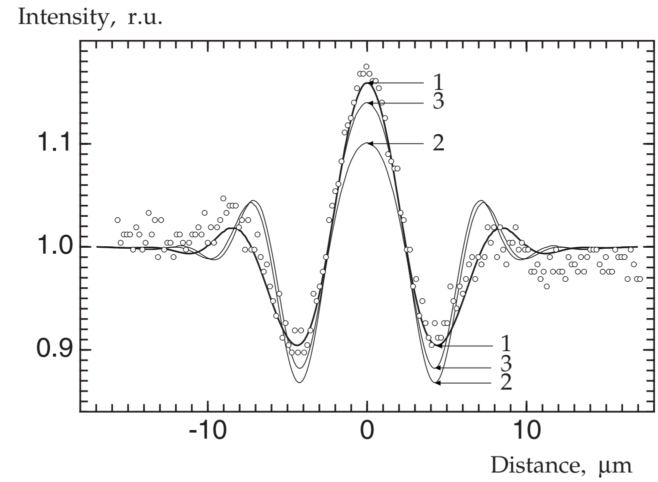

FIG. 2. The experimental (open circles) and simulated (curves 1–3) intensity profiles (the intensity in relative units via the distance across the MP image). The best agreement is achieved through a sequential adjustment in the pipe diameters perpendicular and parallel to the beam. The sample-scintillator distance is 45 cm. The transverse/longitudinal diameters are equal to 5.6/1.0 μm (curve 1, fit$=1.92\times10^{-4}$). For comparison, curve 2 shows the best fit (8.12×10^{-4}) for the circular cross section 1.76/1.76 μm, while curve 3 is given for the intermediate case 3.0/1.4 μm (fit$=5.71\times10^{-4}$).

1.0 μm for MP1 and 0.5 μm for MP2 in Fig. 1). Interestingly, one can see a correlated reduction in the transverse cross sections of the MPs in Fig. 1(c). Here the growth direction was from right to left. Specifically the transverse cross section drastically decreases on the length interval from 329 to 280 μm for MP1 while the same behavior occurs a little later from 89 to 58 μm for its neighbor, MP2. Since the cross section of a MP is tightly related to its Burgers vector,[23] this effect can be explained by the corresponding changes in the Burgers vectors that need appropriate dislocation reactions.

Up to now, it has been generally accepted that any reaction between MPs requires their direct contact, enabling MP dislocations to react with each other. Analytic and computer models of such reactions (hereafter called contact reactions) have been suggested in Refs. 14, 18, 19, and 24. An alternative for contact reactions is contact-free reactions that do not require a direct contact between MPs. These reactions can be realized in two ways. In the first way, one of the two interacting MPs emits a full-core dislocation that moves to the other MP and is accepted by it. In the second way, both MPs emit dislocations that move to each other and react. Although we cannot provide direct experimental justifications of this mechanism, it can explain the above experimental observation of correlated reduction in the cross sections of two neighboring MPs. Indeed, when a MP emits a dislocation, the magnitude B of the MP dislocation Burgers vector decreases by the magnitude b of the emitted dislocation Burgers vector $B'=B-b$. Following Frank,[23] the MP radius R should reduce too from $R\sim B^2$ to $R'\sim(B-b)^2$. On the other hand, when a MP accepts a dislocation of the opposite sign, both its Burgers vector and radius must decrease due to the same rule. Therefore, if two neighboring MPs demonstrate correlated reduction in their radii, one can suppose that, first, these MPs contain dislocations of opposite signs, and, second, they have remotely reacted through the exchange of a full-core dislocation: one of the MPs has emitted this dislocation while the other has accepted it.

As we have already mentioned above, there is a delay between the size reduction in MP1 and MP2. We believe that it is related to the time required by the dislocation to travel between two MPs while the crystal is growing. From this delay ($l\approx0.1$ mm), the crystal growth rate ($v_g=0.5$ mm h^{-1}) and the distance between the MPs ($d\approx0.12$ mm), one can estimate the time of dislocation travel between the two MPs ($t=l/v_g\approx12$ min) and the dislocation velocity ($v_d=d/t\approx0.6$ mm h^{-1}).

Let us perform a theoretical analysis of the necessary conditions for contact-free reactions between MPs. Recently the possibility for the split of a dislocation from a MP at the crystal growth front has been analyzed within a three-dimensional model.[25] Here we consider a simplified two-dimensional model of a contact-free reaction between two parallel MPs. It is intuitively clear that the effect can occur with the MPs having elliptical cross sections like those demonstrated in Fig. 1. However, for the sake of simplicity we consider MPs with circular cross sections of the radii r_1 and r_2 that contain screw dislocations with the Burgers vectors $\mathbf{b}_1$ and $\mathbf{b}_2$, respectively. The distance between the MP axes is denoted as d. Let the first MP emits a screw full-core dislocation with the Burgers vector $\mathbf{b}_0$ [Fig. 3(a)]. To analyze the possibility of such a split event, we utilize the energy variation ΔW associated with dislocation emission. It is calculated (per unit dislocation length) using the previous results[24,26] for the stresses and energies of dislocations lying in an isotropic

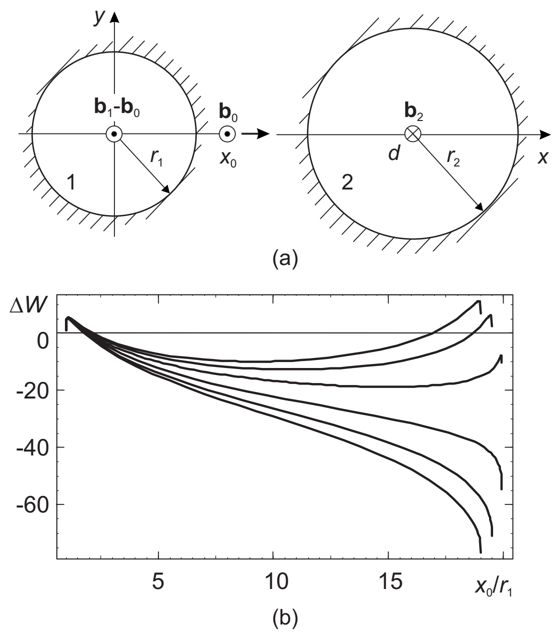

FIG. 3. (a) A model of contact-free reaction between two MPs realized through screw dislocation exchange. MP1 emits a dislocation with the Burgers vector $\mathbf{b}_0$, which moves to MP2, and is absorbed by it. (b) Dependence of the energy ΔW associated with the emission of a dislocation by a MP near a second MP on the normalized dislocation coordinate x_0/r_1 for $d/r_1=20$, $b_1/b_0=7$, and $b_2/b_0=-7,-5,-2,2,5,7$ (from bottom to top). The energy ΔW is given in units of $Gb_0^2/4\pi$.

medium with two cylindrical voids. We obtained an exact formula for ΔW. However, since it is too cumbersome for the present paper, we give below only some results of its numerical examination.

In the following calculations, we will use the Frank relation[23] $r_k=Gb_k^2/(8\pi^2\gamma)$ between the Burgers vector magnitudes b_k and MP radii r_k $(k=1,2)$, where G is the shear modulus and γ is the surface energy. Numerical evaluation of ΔW for the case of growing 6*H*-SiC crystal with $\gamma/G=1.4\times10^{-3}$ nm (Ref. 27) and equilibrium MPs (for which the Frank relation[23] is valid) shows that ΔW depends primarily on the Burgers vectors of MP dislocations [Fig. 3(b)]. As seen in Fig. 3(b), the dislocation exchange is most energetically favorable if the Burgers vectors $\mathbf{b}_1$ and $\mathbf{b}_2$ are opposite in sign. At the same time, to move from one MP to the other, the emitted dislocation must overcome an energetic barrier.

If the Burgers vectors of the two MPs are of the same sign, the emitted dislocation must overcome two energetic barriers on its way from one MP to the other [Fig. 3(b)]. In this case, the possibility for the dislocation exchange between these MPs is governed by the difference in the Burgers vector magnitudes. If $(b_1-b_2)<3b_0$, then we have $\Delta W>0$, and the dislocation exchange is impossible. If $(b_1-b_2)\geq3b_0$, then the dislocation reaction can occur if the emitted dislocation is able to overcome the two energetic barriers. The presence of two energetic barriers results in the appearance of an equilibrium position for the emitted dislocation situated in between the MPs.

In conclusion, we, for the first time, demonstrated a correlated reduction in MP cross sections in the crystal growth of silicon carbide. The effect was found by computer simulating phase contrast images. Our theoretical model shows that the effect can be explained by the exchange of full-core dislocations. This model helps in understanding the mechanisms that control the MP density in bulk SiC crystals. However, the existence of additional mechanisms cannot be ruled out.

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