ISSN 1063-7745, Crystallography Reports, 2016, Vol. 61, No. 6, pp. 914–917. © Pleiades Publishing, Inc., 2016.
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DIFFRACTION AND SCATTERING OF IONIZING RADIATIONS

Study of a Macrodefect in a Silicon Carbid Single Crystal by Means of X-Ray Phase Contrast

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Received September 24, 2015

Abstract—The morphology of a macrodefect in a single-crystal silicon carbide wafer has been investigated by the computer simulation of an experimental X-ray phase-contrast image. A micropipe, i.e., a long cavity with a small (elliptical in the general case) cross section, in a single crystal has been considered as a macrodefect. A far-field image of micropipe has been measured with the aid of synchrotron radiation without a monochromator. The parameters of micropipe elliptical cross section are determined based on one projection in two directions: parallel and perpendicular to the X-ray beam propagation direction, when scanning along the pipe axis. The results demonstrate the efficiency of the phase contrast method supplemented with computer simulation for studying such macrodefects when the defect position in the sample volume is unknown beforehand.

DOI: 10.1134/S1063774516040027

INTRODUCTION

Synchrotron radiation with a photon energy of 10 keV or higher provides images of large defects (voids) in a single crystal, with minimum sizes from tenths of a micrometer to several micrometers, due to the change in the X-ray wave phase shift, because the phase shift per beam path length unit in the object changes the wave field faster than the absorption does by a factor of 100 or even more. The phase contrast, as well as the absorption contrast, can be measured using simple transmission geometry [1, 2] by recording radiation at some distance from the object.

For large objects with sizes of several tens of micrometers or more, which efficiently deviate X rays at the boundaries due to the refraction, images can be obtained by the phase contrast method even at short distances from the object (i.e., in the near field) and under the conditions of low temporal coherence [3]. Moreover, the relatively wide spectrum of synchrotron radiation is used to study the internal structure by tomography [4] and phase radiography [5, 6]. It should be noted that 3D images are obtained in tomography by the direct method without applying the diffraction theory, which is often not quite correct.

On the other hand, images of microscopic objects with a cross section about 1 μm in size can be observed only at large distances from the detector (i.e., in the far field), because both the contrast and image size at short distances are too small (below the detector resolution). With an increase in distance, a small object forms a diffraction pattern with a higher contrast and larger image size; however, in this case, in order to determine the real sizes of a microscopic object, one must solve the inverse problem by carrying out computer simulation of (see, e.g., [7]) an experimental image.

In this paper we report the results of studying micropipes, i.e., cavities in a silicon carbide single crystal in the form of pipes with elliptical cross section. A quantitative study of their morphology (i.e., determination of their orientation and the cross section shape and sizes) is of considerable interest. The inverse problem is significantly simplified because the change of the object density along the micropipe axis can be neglected in this case.

It is known that the radius of the first Fresnel zone is $r = (\lambda z)^{1/2}$, where z is the distance from the object to the detector and λ is the wavelength. If r is much smaller than the lateral object sizes, the details of its cross section can be reconstructed from a series of 2D projections measured during sample rotation (tomography). However, the micropipe position in the volume of a platelike crystal is unknown beforehand; hence, tomography cannot be used in this case.

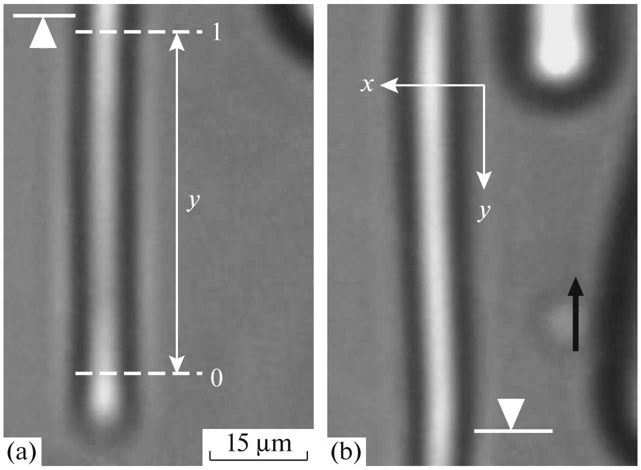

Fig. 1. Phase-contrast images of the same micropipe in the bulk of 4*H*-SiC crystal: (a) at the beginning of the pipe and (b) along its growth direction. The fragments are aligned at the level indicated by a white arrow. Levels 0 and 1 limit the image simulation range: $y = 51.8$ μm. The crystal growth direction is shown by a black arrow. Distance $z = 45$ cm.

Here, the change in the cross section along the micropipe axis occurs at distances larger than r; therefore, the object can be considered homogeneous along the pipe axis in each cross section and, to solve the inverse problem, it is sufficient to use only the intensity distribution across the micropipe in one projection. In this case, computer simulation of the experimental intensity distribution for a specific micropipe elliptical cross section makes it possible to determine two parameters of this cross section. Moving along the micropipe axis, one can also determine the changes in these parameters based on one 2D projection.

It was established in earlier papers [8, 9] that a micropipe in a silicon carbide single crystal may have an elliptical cross section. In this study we clearly showed for the first time that this cross section may rotate during the crystal growth (i.e., when moving along the pipe axis) without a significant change of the cross-sectional area. This fact can be established based on one 2D projection only by a computer simulation of experimental data. It should also be noted that, as was shown in [7], the calculated diameters of one cross section in the directions perpendicular and parallel to the beam propagation direction cannot be unambiguously compared with the real diameters of micropipe elliptical cross section. However, when the elliptical micropipe cross section rotates, these diameters are determined unambiguously as the minimum and maximum diameter values from the entire dataset. Accordingly, one can determine the rotation angle.

EXPERIMENT AND COMPUTER SIMULATION

Experimental images of micropipes were obtained on the synchrotron radiation source of intermediate brightness: Pohang Light Source (Southern Korea), without a monochromator. On the 6D (X-ray microimaging) beamline, the radiation from the bending magnet (located at distance of 32 m from the sample) was used; the vertical source size was 60 μm. Due to the small angular size of the source (2×10^{-6} rad), the spatial coherence length in the illuminated region of the object is 42 μm, which is much larger than the sizes of the micropipe cross section in a crystal.

Far-field images were obtained using a high-resolution detector sensitive to visible light. X rays passed through the sample to excite fluorescence in a $CdWO_4$ scintillator crystal. Then an optical objective lens 20× focused the image onto the detector matrix with a resolution of 1600 × 1200 pixels, effectively reducing the pixel size from 7.4 to 0.37 μm.

Samples in the form of plates were cut along the growth direction of 4*H*-SiC boule and carefully polished from both sides. Micropipes are defects of crystal structure, which are formed during SiC crystal growth [10]. They are generally oriented parallel to the growth direction [0001]. The micropipe axes (directed parallel to the sample surface) were oriented perpendicular to the beam propagation direction, to lie in the horizontal plane in order to use the smallest (vertical) source size. Phase-contrast images (Fig. 1) were obtained using a beam parallel to the z axis. It is rotated by an angle of 90° to make the micropipe (directed along the y axis) lie in the vertical plane. Experimental intensity profiles were measured across the micropipe axis (along the x axis) using the ImageJ program [11].

Numerical simulation was performed with the aid of the FIMTIM program, which was described in detail in [7–9, 12–15]. The micropipe cross section by the beam was assumed to be elliptical. The directions of the transverse D and longitudinal D_1 cross-section diameters are oriented, respectively, perpendicular and parallel to the beam axis. The program calculates theoretical intensity profiles, automatically changing parameters D and D_1, and compares them with the experimental intensity profile. The wide emission spectrum was taken into account in the calculation by summing images for monochromatic harmonics with a weight corresponding to the real synchrotron radiation spectrum measured by a detector. The program calculates the emission spectrum taking into account all the absorbers on the beam path, including the sample.

During the calculation, the program automatically searches for the nearest minimum of the sum of squared deviations $\chi^2(D, D_1)$ of the theoretical profile from the experimental points, beginning with the initial values on the square grid of D and D_1 values, gradually decreasing the grid step. To estimate the error in calculating the diameters of the micropipe cross section at the minimum point $\chi^2(D, D_1)$, the program calculates a two-dimensional map of function

$\chi^2(D, D_1) - \chi^2_{\min}$ in the vicinity of this point. The sharper the minimum in any direction is, the more accurately the cross section diameter is determined.

RESULTS AND DISCUSSION

Figure 1 shows two images of one micropipe: the first at its beginning and the second at some distance along its growth direction. In the lower part of the image in Fig. 1a, the contrast is bright at the center of the pipe and dark at its periphery; the micropipe center is darker in the middle part of the image and bright again in the upper part of the image. Above the level indicated by a white arrow in Fig. 1b, the micropipe axis deviates from the crystal growth direction, first to the left and then to the right side. Similar deviations were observed for many other micropipes in SiC crystals of 4*H* and 6*H* polytypes grown by the sublimation method.

In addition, the transverse sizes of micropipes changed along their axes. One can suggest that the change in the contrast in Fig. 1a is caused by changes either in the cross-sectional area, in the shape of the cross section, or in both parameters. When the one-dimensional model is used, the image structure should not change at a distance shorter than $2r$ along the micropipe axis (r is the radius of the first Fresnel zone). In other words, the one-dimensional model is applicable only when changes along the axis occur sufficiently slowly. This condition was satisfied in our case.

Experimental intensity profiles were measured with a small step within a segment of length $y = 51.8$ µm (Fig. 1), which is much larger than the radius of the first Fresnel zone $r = 5.9$ µm, at a distance of $z = 45$ cm from the sample. According to the results of computer simulation of the cross sections between the 0 and 1 levels, the cross-sectional area σ changed only slightly; however, diameters D and D_1 changed significantly. It is reasonable to suggest that the cross section rotated around the micropipe axis. To determine the rotation angle, the small change in the area was excluded by dividing D by ratio σ/σ_1, where σ_1 and σ are the cross-sectional areas at the beginning and at the current point in the interval under consideration. As a result, the dependence of the rotation angle of diameters on the distance along the micropipe axis was obtained. Figure 2a presents curves $D(y)$ and $D_1(y)$ in black and gray colors, respectively. The behavior of these curves confirms that the micropipe rotated twice around its axis by 90°. The micropipe rotation is accompanied by very small changes of its cross-sectional area σ. It is of interest that the first rotation (near the nucleation site) occurred more rapidly than the second one.

Let rotation angle φ be zero at level 1. Then $d = D$ and $d_1 = D_1$. At the other points, we have

$$D = [(d\cos\varphi)^2 + (d_1\sin\varphi)^2]^{1/2}, \quad D_1 = dd_1/D. \quad (1)$$

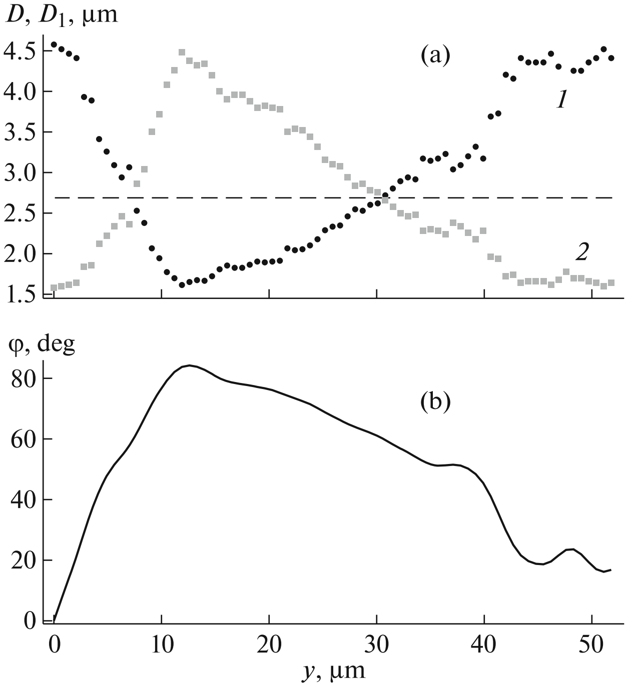

Fig. 2. (a) Dependence of the (*1*) transverse $D(y)$ and (*2*) longitudinal $D_1(y)$ diameters of the micropipe cross section on distance y along the micropipe axis. Micropipe cross-sectional area σ is presented by the value $(DD_1)^{1/2}$ and is shown by a dotted straight line. (b) Dependence of rotation angle φ of the elliptical cross section on distance y.

This equation makes it possible to determine the dependence of rotation angle φ on distance y in the direction from D to D_1 from the formula

$$\sin\varphi = \left[(1 - S^2/d^2)/(1 - d_1^2/d^2)\right]^{1/2}, \quad (2)$$

assuming first that $S = D$ and then that $S = dd_1/D_1$. Figure 2b shows the dependence $\varphi(y)$. The curve is asymmetric, which clearly indicates the double rotation of the elliptical cross section (from 0° to 90° and from 90° to 0°) with different rates.

CONCLUSIONS

The computer simulation of the experimental intensity distributions in the direction perpendicular to the beam and micropipe axes, based on one 2D-projection, made it possible to determine the size and shape of the micropipe cross section in the bulk of SiC single crystal and reveal the change in these parameters during crystal growth. The wide synchrotron radiation spectrum and simple geometry in transmission used in our experiment provided high intensity but did not allow us to observe the diffraction patterns at all points, except for the central part of the micropipe image. Nevertheless, even simple intensity profiles can be used for simulation. The parameters characterizing the micropipe cross section in the directions parallel and perpendicular to the beam propagation direction when moving along the micropipe axis were obtained as a result. Note that the cross-section rotation angle φ cannot be determined by the direct method because the micropipe position in the sample is unknown beforehand.

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Translated by Yu. Sin'kov