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Bright-field imaging of lattice distortions using x rays

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Can x rays yield bright-field images of crystalline systems similar to those of transmission electron microscopy? So far, the response was negative, but the authors present here a positive case: bright-field x-ray images carrying information both from diffraction/scattering phenomena and from absorption and phase contrast. Specifically, synchrotron x-ray transmission micrographs simultaneously yielded diffraction-based information on strain effects and information on structural inhomogeneities when (0001) 4*H*-SiC wafers were set for a strong reflection in the Laue geometry. This approach offers interesting advantages with respect to the separate study of strain and inhomogeneity effects for a variety of crystalline systems. © *2006 American Institute of Physics*.
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Bright-field (BF) images[1] are well known in transmission electron microscopy (TEM): images created by unscattered electrons are entirely due to mass-thickness variations in amorphous samples but may include diffraction contrast in crystalline samples. On the contrary, no x-ray images that carry diffraction-created information and standard radiographic information were so far obtained.[2] We used here collimated monochromatic x rays to obtain radiographs similar to bright-field TEM images with both kinds of information, specifically revealing strain-related lattice distortions in SiC.

In a conventional radiograph, contrast is due to absorption and—under certain conditions[3–5]—to phase gradients. Here we show that when (0001) 4*H*-SiC wafers are set for a strong reflection at the Laue geometry, images yielded by the transmitted beam also exhibit reversed topographic strain contrast features caused by local depletion of x rays due to Bragg diffraction.

The experiments were performed at the XOR 2-BM beamline of the Advanced Photon Source in the Argonne National Laboratory. The experimental setup is shown in Fig. 1(a): an energy tunable monochromatic x-ray beam (bandwidth $\Delta\lambda/\lambda \approx 1.5\times 10^{-4}$) was provided by a Si (111) double-bounce monochromator. Samples of (0001) 4*H*-SiC wafers (0.3–0.45 mm thickness, 30 mm diameter) were prepared from different crystals grown by the sublimation method. High-resolution transmission images[6] were obtained by converting x rays into visible light with $CdWO_4$ scintillation crystal and then focusing the light into the 1000×1000 charge-coupled device (CCD) chip through a 20× objective lens, reaching a 0.65 μm effective pixel resolution. The CCD detection system was placed 200 mm away from the sample to obtain radiographic phase contrast in transmission images.[3–5] By detecting the diffracted beam, we also obtained the equivalent of dark-field (DF) TEM images; in this case, the CCD system was placed 5 mm from the sample to enhance the spatial resolution.

In principle, any sample position angle θ and x-ray photon energy E could produce diffraction effects in the transmission (BF) images. In practice, we could only detect them after adopting two conditions: (1) a single reflection with strong intensity and (2) perfect in-plane (vertical to the wafer surface) reflections. Theoretical simulation and empirical

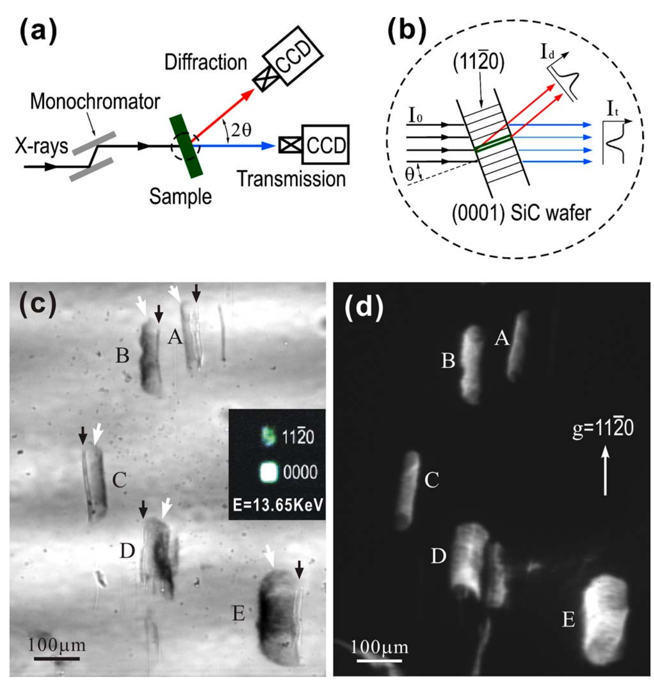

FIG. 1. (Color online) X-ray bright-field images of SiC simultaneously reveal hollow-tube micropipes and related lattice displacements. (a) Experimental setup. (b) Schematic illustration of contrast inversion between transmitted (BF) and diffracted (DF) beams. (c) BF image; the inset is the Laue pattern showing the 11$\bar{2}$0 reflection. (d) DF image.

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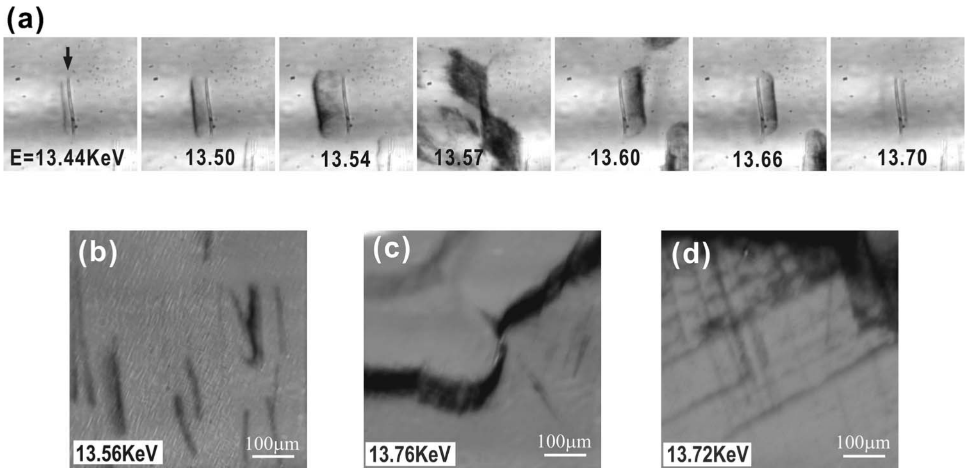

FIG. 2. (a) BF images for different photon energies E showing displacements of the $11\bar{2}0$ lattice plane around a micropipe (the black arrow). [(b)–(d)] BF images showing lattice distortions associated with micropipes with full core, lattice plane bending, and grain boundaries.

tests led us to select the $11\bar{2}0$ plane as the optimal reflection for BF imaging in SiC.

Figure 1(c) shows an example of BF imaging under the above optimal conditions ($E=13.65$ keV, $\theta=17°$); the Laue pattern in the inset demonstrates the strong reflection. The black arrows identify micropipes[7] (hollow-tube superdislocations[8,9]) penetrating through the wafer thickness. Border enhancement by phase contrast[3–5] makes these very small (0.1–1 μm in diameter) features clearly visible.

Additional features marked by the white arrows are explained instead by diffraction since they are dark (diffraction-caused loss of intensity) and drastically change with E. This interpretation is validated by the DF image of the same reflection in Fig. 1(d). We see a clear correspondence and complementary contrast of the BF diffraction features and the DF features.

Thus, even without DF imaging, BF radiographs can be used to study lattice distortions. This approach offers relevant advantages with respect to diffraction (DF) imaging. First, it provides information both in the real and in the reciprocal space, making it possible to reveal correlations, whereas this is not feasible with DF images. The information delivered by DF micrographs is in general more limited. For example, they cannot reveal structural inhomogeneities. Furthermore, our approach allows a one-to-one correlation between sample and image, while in DF imaging the correlations can be disturbed by overlap and/or separation of diffracted x rays with different directions.[10]

We performed preliminary tests of our BF imaging to identify different types of lattice defects. Figure 2(a) shows the E dependence of the dark features revealing the spatial distribution of $11\bar{2}0$ lattice planes around a micropipe [C in Fig. 1(c)]. This approach could quantitatively study aspects of the local dislocations[8,9] during micropipe formation such as strain field magnitudes, the Burgers vector sign, the hollow-core size, etc. Figure 2(b) shows dark-contrast features with no hollow-tube image, due to full-core micropipes.[11] The long dark-contrast feature (bend contour[1]) in Fig. 2(c) reveals the existence of lattice plane bending, and its E dependence can be used to estimate the type and direction of the bent. The orthogonal network in Fig. 2(d) shows grain boundaries probably caused by large lattice plane bending during the crystal growth.

This is, in summary, the demonstration of TEM-like BF diffraction contrast in radiology. Note, in particular, the difference with respect to the approach of Davis *et al.*[12] which was not related to Bragg diffraction. Following the results described here, our technique can be applied to a variety of crystalline systems to simultaneously study structural inhomogeneities such as micropipes or voids and local lattice distortions due to strain fields, mosaicity, or grain boundaries.

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