Weak-Beam Method in X-Ray Topography and Observation of Threading Dislocations in 4H-SiC

Dislocations in silicon carbide (SiC) have been observed by means of the weak-beam technique of X-ray topography with Bragg-case geometry. Using an X-ray beam with a dispersion of 0.87 μrad collimated by an asymmetric Si 331 reflection, a nearly intrinsic rocking curve of the SiC 0008 reflection was obtained. High-resolution contrast images of threading screw dislocations were obtained by kinematical diffraction at angles of ±24 μrad from the rocking curve peak, revealing their depth profiles inside the wafer, and the characteristics of their sense. The technique is equivalent to weak-beam dark field imaging in electron diffraction.

Dark-field imaging X-ray topography is a technique for observing defects in a crystal with high-sensitivity. Synchrotron radiation X-rays give clear and high-resolution images owing to their brilliant and parallel nature. X-ray topographs for high-quality crystal-like semiconductor wafers at the Bragg angle suffer from intense reflections from perfect-crystal regions, and dynamical effects. The dislocation contrasts, therefore, become complicated, and the core dislocation regions are invisible. On the other hand, under conditions deviating from the Bragg angle, the heavily-distorted regions near to the dislocation core satisfy the Bragg condition whereas the diffraction intensity from the perfect regions decrease, thereby generating high resolution and highly sensitive contrasts. This technique is known as the weak-beam method, for which experimental and analytical methods have been well investigated in electron diffraction dark-field imaging [1]. In X-ray topography, the analogous weak-beam method has been studied, and high-resolution dislocation images were observed [2-4]. In this study, we have analyzed dislocation contrasts in detail using weak beam X-ray topography for the Bragg-case geometry [5]. In order to obtain a clear weak-beam contrast, the incident beam was collimated so that its angular divergence was well within the angular width of the rocking curve of the diffraction from the specimen.

The specimen studied was a wafer of 4H-SiC [6], which is a wide-gap semiconductor extensively studied for use in high-efficiency, energy-conserving power devices [7]. Since the wafers suffer from a fairly large density of dislocations, dislocation analysis is important for developing SiC-based devices. Threading-screw dislocation is a typical dislocation ocurring in SiC, penetrating along the <0001> direction of the crystal with a Burgers vector ***b*** = <0001>. These dislocations are likely to cause device failures, such as current-leakage under reverse-biased conditions, for example.

The incident beam was collimated by an asymmetric Si 331 reflection (interplanar spacing d = 0.124575 nm) from the Si (111) surface, and the 0008 reflection of 4H-SiC (d = 0.125625 nm) was observed (Fig. 1). For a wavelength of 0.097 nm, the Darwin width of Si 331 reflection, 0.87 μrad, is sufficiently smaller than that of the SiC symmetric 0008 reflection, 6.06 μrad.

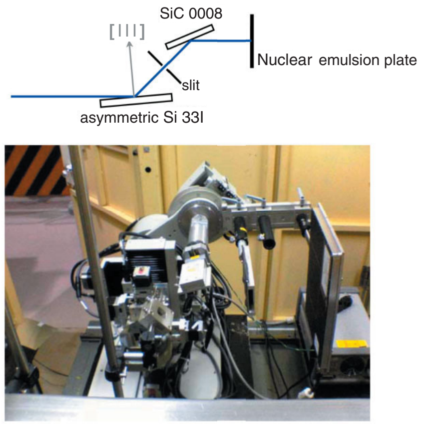

Figure 1
Experimental setup. Monochromatic X-rays from the double-crystal monochromator are collimated by an asymmetric Si 331 reflection, and the X-ray topograph is recorded on a nuclear emulsion plate.

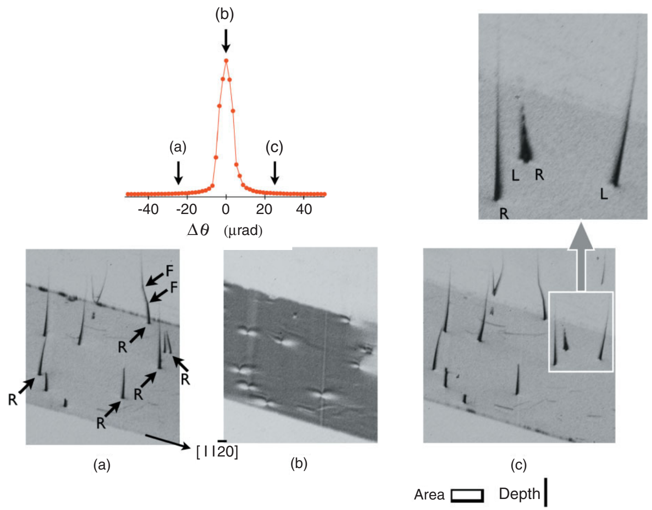

Figure 2
Rocking curve of the 4H-SiC 0008 reflection, and X-ray topographs taken at the angles (a), (b) and (c) in the rocking curve. The scale of the topograph is shown by the rectangle and the vertical bar: the rectangle represents an area of 0.1 mm × 0.1 mm, and the vertical bar represents a depth of 0.1 mm. In topograph (a), the screw dislocations designated by "R" are right-handed, and the rest are left-handed. Arrows labeled with "F" indicate folding points in screw dislocations. Part of (c) is shown on a magnified scale. "L" and "R" denote respectively right-handed and left-handed screw dislocations.

Figure 2 shows a typical set of X-ray topographs, taken at the peak of the rocking curve and at lower and higher angle positions deviating from the peak by 24 μrad. At the peak of the rocking curve, intense reflection comes from the nearly-perfect crystal region, but isolated bright regions, indicating missing diffraction intensity, are also found. These come from local strain at outcrops of threading-screw dislocation. At angles ±24 μrad from the rocking-curve peak, completely different images are observed. Since the high-intensity reflection from the wafer surface is eliminated under these conditions, the weak diffraction contrast from the distorted regions in the vicinity of dislocation become visible over the lowered background reflection. The threading dislocations are observed as shapes elongated towards the inside of wafer, with attenuation in intensity due to absorption by the specimen. We also found some dislocations folding inside the wafer [Fig. 2(a)], which may occur due to stress induced during the crystal growth process. In weak-beam images taken for other wafers, we observed further cases in which a dislocation bends or merges with basal-plane dislocations. Another feature of the dislocation contrast is an indication of the sense of a screw dislocation. The misorientation angle of the diffraction plane is bilaterally antisymmetric about the axis of a threading screw dislocation. It is positive (negative) on the left-hand (right-hand) side for a right-handed screw dislocation, and vice versa in the left-handed case. Therefore, the sense can be determined straightforwardly from the contrast, as shown in Fig. 2(a) and (c).

In this study, the weak-beam image is obtained clearly by using a collimated X-ray beam. Analysis is possible using only kinematical considerations, and the dislocation nature can be analyzed using the same criteria as for weak-beam electron diffraction imaging. In addition, the Bragg-case geometry is found to be useful for cross-sectional analysis near the upper face of a wafer, but it does not require any samples to be destroyed.

REFERENCES

[1] D.B. Williams and C.B. Carter, *Transmission Electron Microscopy*, (Plenum Press, New York, 1996) Chap. 26.
[2] J.F. Petroff, M. Sauvage, P. Riglet and H. Hashizume, *Phil. Mag.*, **A42** (1980) 319.
[3] R. Barrett, J. Barushel, J. Härtwig and F. Zontone, *J. Phys. D: Appl. Phys.*, **28** (1995) A250.
[4] M. Koishi, N. Ohya, Y. Mukobayashi, H. Koizumi, K. Kojima and M. Tachibara, *Cryst. Growth Des.*, **7** (2007) 2182.
[5] H. Yamaguchi, H. Matsuhata and I. Nagai, *Materials Science Forum*, **600-603** (2009) 313.
[6] SiC exhibits more than 250 polymorphs and 4H-SiC is one of the polymorphs that is most extensively studied for power device applications. 4H refers to the four-bilayer periodicity of the stacking with hexagonal symmetry.
[7] H. Yamaguchi, H. Matsuhata, T. Ohyanagi, H. Okumura, K. Arai and H. Shimizu, *Users' Report, PF Activity Report 2008*, **26** (2008) 160.

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