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Evidence for 9R-SiC?

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Abstract: Complementary to our first paper on the origin of threefold contrast on SiC high resolution transmission electron microscopy (HRTEM) images, we now provide an example of threefold contrast produced by a stacking layer sequence which corresponds to one unit cell of the 9R polytype.

Key words: silicon carbide, SiC polytypes, 9R-SiC, high resolution transmission electron microscopy, high resolution image simulation, whiskers

Introduction

SiC is one of the prototypes of polytypic materials and the number of stacking periodicity may range in principle from two to infinity (Baronnet, 1993), however, only a few are commonly observed (2H, 4H, 6H, 3C, and 15R). In previous works (Kaiser et al., 1998, 1999) we described a threefold periodicity in SiC layers which was unambiguously attributed to the overlap of the two cubic SiC twin variants and not as suggested earlier (Jepps and Page, 1980) to the existence of the 9R-SiC polytype. It could be proved by applying a simple criteria (Kaiser et al., 1999) that, in transmission electron microscopy (TEM) images, no evidence for the 9R-SiC polytype has been presented up to now. The purpose of this article is to demonstrate that a 9R stacking really occurs in SiC grown in thermodynamically nonequilibrium conditions, namely in catalytically synthesized SiC whiskers.

Materials and Methods

The SiC whiskers examined were grown by vapor-liquid-solid process promoted by Fe catalyst (Urretavizcaya and Porto Lopez, 1994). Variation of growing conditions led to dominant growth of different SiC polytypes.

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TEM samples were prepared by deposition of whiskers on standard copper grid covered with holey carbon film and then examined using JEM-2010 and JEM-3010 electron microscopes (JEOL). High resolution electron micrographs were recorded along the [11–20] zone axis of the whiskers. The parameters for the 9R-SiC atom model are described in Kaiser et al. (1999). Image processing was performed using Digital Micrograph (Gatan).

Results and Discussion

Figure 1a shows a high resolution transmission electron microscopy (HRTEM) image of the edge of a SiC whisker. Along with regions of 2H SiC (in Hägg notation [+-+-+- . . .]) with a spacing of 0.5 nm (marked), regions showing a threefold periodicity are observed in the whisker. Always three periods of the threefold contrast are seen simultaneously making a 2.25 nm spacing. This leads to the assumption that the stacking sequence can be described in Hägg notations as [++-++-++-] which matches one unit cell of the 9R polytype. In Figure 1b, the boxed area of Figure 1a is enlarged. The atom model of the 9R-SiC unit cell is overlaid, and the accurate fit demonstrates that this cell is the base of the lattice image seen. The fast Fourier transform (FFT) pattern of Figure 1b is seen in Figure 1c showing no reflection perpendicular to the (0001) planes that additionally proves the existence of the 9R SiC structure (see Kaiser et al., 1999).

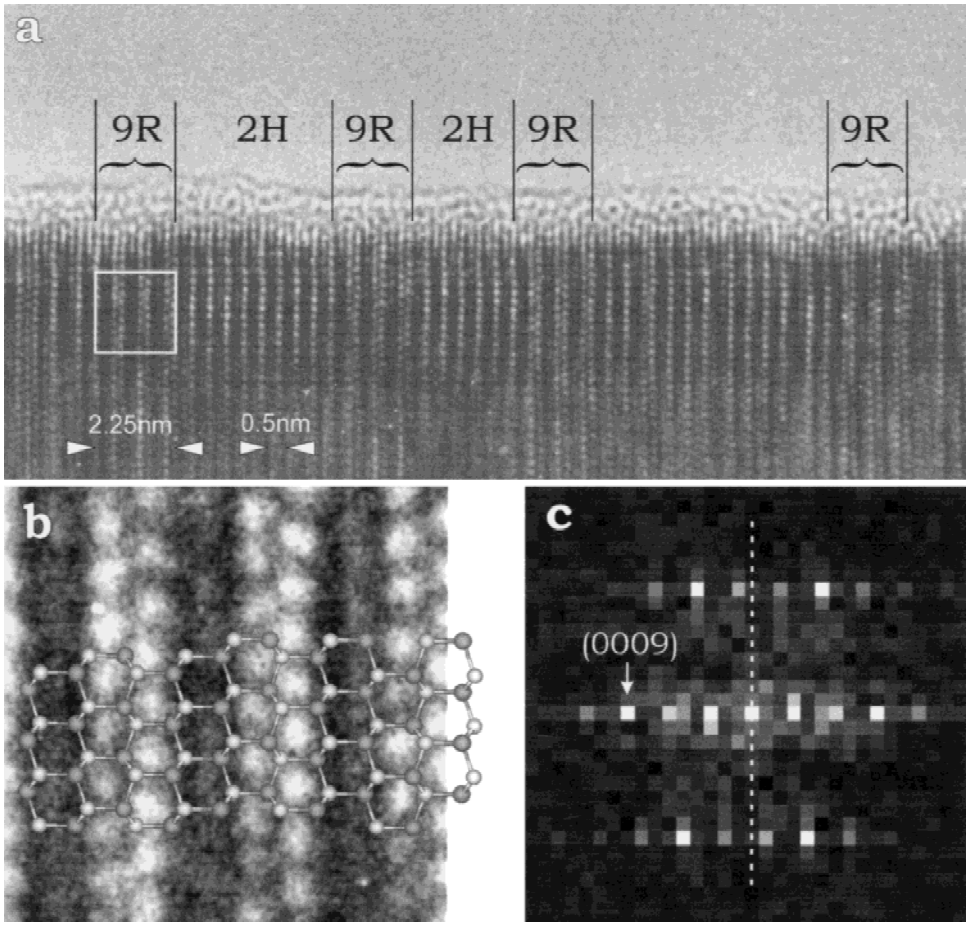

Figure 1. a: High resolution transmission electron microscopy (HRTEM) image of 2H-SiC whisker at the [11–20] zone. Three regions exhibiting a threefold periodicity with a spacing of 2.25 nm (arrowheads) spread repeatedly through the whole whisker, and broader regions of plain 2H contrast (marked) are seen as well. **b:** The enlarged area of the boxed area in panel a overlaid with the structure projection of the 9R-SiC polytype. In **c,** the fast Fourier transform (FFT) pattern from the experimental image in panel b is shown to demonstrate that there is no reflection perpendicular to the 0009 direction.

Generally, the variation in the polytype microstructures can be described in terms of regular stacking faults. It is interesting to note (see Fig. 1a) that always only one 9R unit cell appears after a varying number (6–12) of repetitions of the 2H-SiC polytype. Of course, it is not possible to declare one structural period as a 9R crystal phase. Further work will address the problem of understanding the stacking sequence creation described.

Summary

Contrary to the threefold contrast observed previously in HRTEM images of cubic SiC (Kaiser et al., 1999; Jepps et al., 1979) originating from the overlap of twins, we showed in this article that the threefold contrast observed in HRTEM images of 2H-SiC whiskers can be attributed to the 9R-SiC stacking sequence.

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