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Application of Variable C_S HRTEM to the Study of Nanoscale Structures

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As nanoscale systems are increasingly incorporated into industrial products, an understanding of defect behavior and interfaces in these materials is critical. Because of the inherently small dimensions of the "nano" regime, characterization of nanoparticle-based systems demands high resolution. The transmission electron microscope (TEM) is uniquely suited for this task, although its resolution is limited by the aberrations present in electromagnetic lenses [1].

The incorporation of aberration-corrected lenses in the TEM can provide many advantages for microstructural and chemical analysis [2-4]. The use of variable C_S HRTEM allows the study of surfaces and interfaces with improved contrast at a low level of image delocalization, electron holography, or other means to remove aberrations numerically. This study utilizes a Philips CM200 FEG with a computer-controlled electromagnetic hextapole system for the correction of spherical aberration of the objective lens [5, 6]. A point resolution better than 0.13 nm is available in this instrument, and the blurring of high-resolution information by the phenomenon of contrast delocalization is minimized [3]. The TEM is used for the recording of defocus series of images, which aid the analysis of defocus levels and structure imformation; images were recorded using a 2k × 2k CCD camera binned to 1k × 1k format.

To illustrate the potential of this instrument for the analysis of nanoscale systems different forms of crystalline Si and β-SiC nanoparticles were analyzed. Figure 1 is a HRTEM image of a representative β-SiC nanosphere supported on a carbon film viewed along [011]. This nanoparticle exhibits nanotwins with twin boundaries, which are atomically flat across the diameter of the particle, and an amorphous surface layer. Similarly, the image of a Si nanoparticle viewed along [100] reveals an amorphous oxide layer of irregular thickness (Figure 2). Conventional HRTEM imaging often superimposes image contrast of oxide layers on a nanoparticle with image contrast of the amorphous support film, making the thickness of the oxide layer difficult to determine. However, the use of aberration-corrected imaging allows the contrast from the support film to be minimized and the thickness of the oxide to be measured.

A relatively difficult task is the identification of nanocrystalline inclusions in an amorphous film. Figure 3 is a focal series of a silicon nanocrystal that contains a stacking fault. Here, because both the nanocrystal and the amorphous matrix are silicon, there is no difference in chemistry to aid in the analysis. With the capability to tailor the imaging conditions of the TEM for reducing the contrast of the surrounding amorphous material, the nanocrystallites are readily identified. Further, it is apparent that the use of a focal series provides confirmation of the crystallinity of the inclusions and important information that can be compared to a model of the proposed defect structure.

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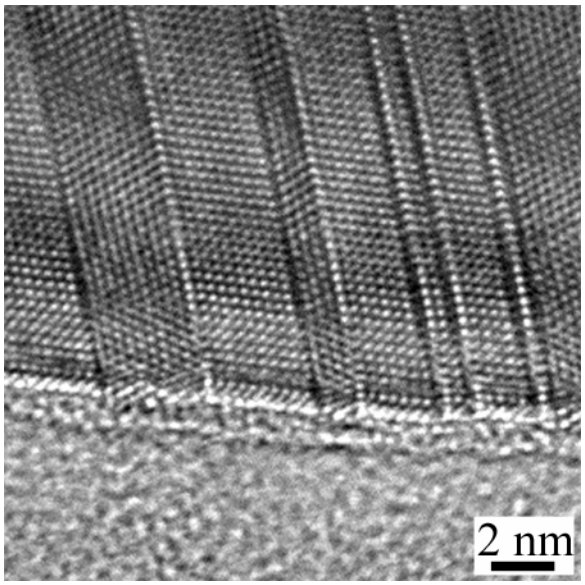

FIG. 1. HRTEM image of a β-SiC nanosphere viewed along [011] that contains {111} nanotwins.

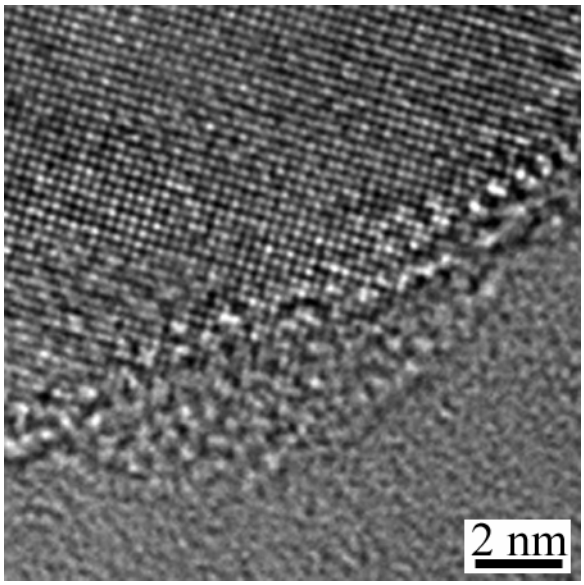

FIG. 2. HRTEM image of a Si nanoparticle with an amorphous surface layer of irregular thickness viewed along [100].

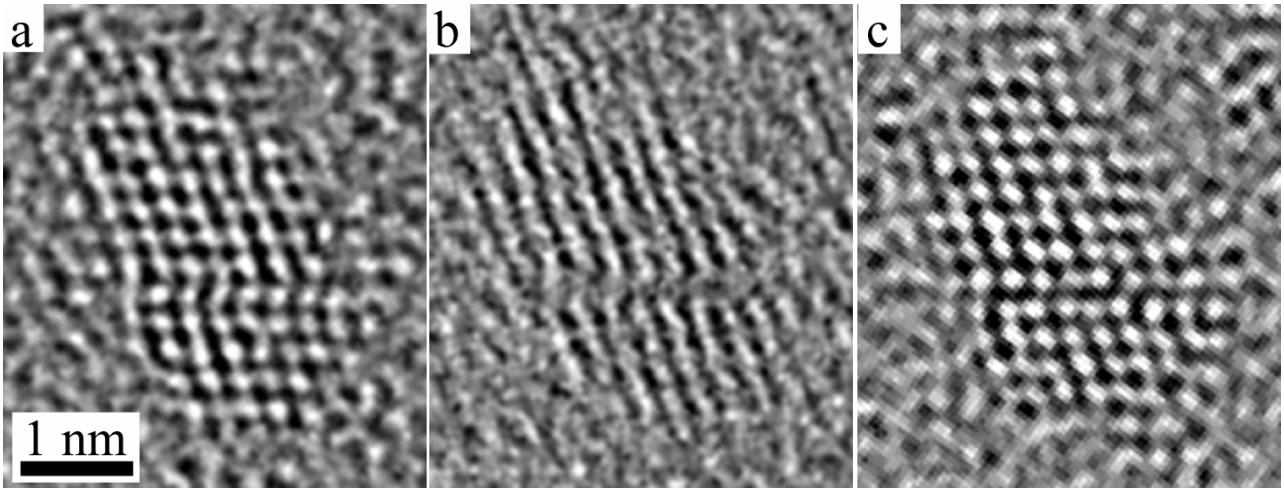

FIG. 3. HRTEM images of a Si nanoparticle containing a stacking fault in an amorphous Si film. The images are at defocus values of (a) 9.3 nm, (b) 0 nm, and (c) -21.7 nm showing the phase reversal.

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