doi:10.1017/S143192761005991X
Microsc. Microanal. 16 (Suppl 2), 2010

Surface Channeling in Aberration-Corrected STEM of Nanostructures

Jingyue (Jimmy) Liu* and Lawrence F. Allard**

* Center for Nanoscience, Department of Physics and Astronomy, Department of Chemistry and Biochemistry, UM-St. Louis, One University Boulevard, St. Louis, MO 63121

** Materials Science & Tech. Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831

Sub-Ångström resolution imaging and atomic resolution spectroscopy in aberration-corrected STEM are becoming routinely available, providing information on crystal structure, defect structure, elemental composition, and electronic structure with a sensitivity of, or approaching, individual atoms. To quantitatively interpret image intensities, however, requires a better understanding of the origin of the collected signals. We report here observation of anomalous intensity enhancement in sub-Ångström resolution HAADF images of surfaces and interfaces of ZnO nanostructures. We propose that the significant increase of the collected HAADF signal of a row of atom columns close to the crystal surface originates from a surface-resonance channeling effect [1].

The ZnO nanobelts used in these experiments were fabricated by a thermal evaporation-condensation method in a high-temperature tube furnace. Each nanobelt is a single crystal with relatively uniform thickness and some nanobelts possess atomically flat and clean surfaces. A JEOL 2200FS FEG STEM/TEM with a hexapole aberration corrector (CEOS GmbH, Heidelberg, Ger.) for the probe-forming optics, providing a nominal resolution of 0.07 nm in HAADF imaging mode, was used to obtain the HAADF images. The HAADF detector was operated with inner and outer collection semi-angles of 100 mrad and 170 mrad, respectively.

Figures 1a and 1b show, respectively, a HAADF image and the corresponding BF STEM image of a ZnO nanobelt, oriented along the [11-20] zone axis. The computed diffractogram is shown in Fig. 1b. The amorphous material, clearly revealed in Fig. 1b, originated from the buildup of electron beam-induced contamination. Even with the presence of a thick layer of amorphous material, atomic resolution images were obtained as is clearly shown in the inset of Fig. 1a. In addition to revealing the atomic arrangement of the Zn columns, the most striking feature in the HAADF images is the bright row of atom columns (hereafter, 'bright atom row'), running along the ZnO [-1100] direction, close to the ZnO (0001) surface; the BF STEM image shows a dark band corresponding to the same near-surface atomic columns. To quantify the intensity enhancement of the bright atom row, an intensity profile across the ZnO (0001) lattice fringes within the rectangular box in Fig. 1a, projected along the ZnO [-1100] direction, is displayed in Fig. 1c (Only a portion of the intensity profile is shown). The intensity enhancement of the bright atom row close to the (0001) surface is significant; the fringe visibility of this row was estimated to be about 2.5 times that of the lattice fringes inside the ZnO nanobelts. The spacing between the bright atom row and the next row of Zn atoms within the ZnO crystal was measured to be approximately 0.30 nm, much larger than the ZnO bulk lattice spacing of 0.26 nm. The high magnification HAADF image shown in the inset of Fig. 1a reveals that the bright, near-surface atom plane does not represent the outmost surface atoms of the ZnO (0001) surface. There is at least one layer, and in some places probably two layers, of Zn atoms beyond this bright atomic plane. These layers, however, possess much lower intensities than those of the bulk Zn layers, suggesting that the ZnO (0001) surface has an incomplete growth of the outer-most surface layer. The bright atom row represents the first complete ZnO (0001) layer near the surface. These observations and a series of other experiments suggest that a surface-resonance channeling effect may play a major role in determining the observed anomaly of intensity enhancement of HAADF images of structurally perfect surfaces [1]. A better understanding of the effect of surface/interface channeling on HAADF/ADF image contrast may have implications for the quantitative interpretation of those images and may provide new routes to utilization of this technique to study surface structures with sub-Ångström resolution [2].

References

1 J.M. Cowley, *Ultramicroscopy* **27** (1989) 319.

2 This research is supported by the University of Missouri-St. Louis; the electron microscopy work was conducted at the Oak Ridge National Laboratory's High Temperature Materials Laboratory, sponsored by the U.S. DOE, Office of Energy Efficiency & Renewable Energy, Vehicle Technologies Program.

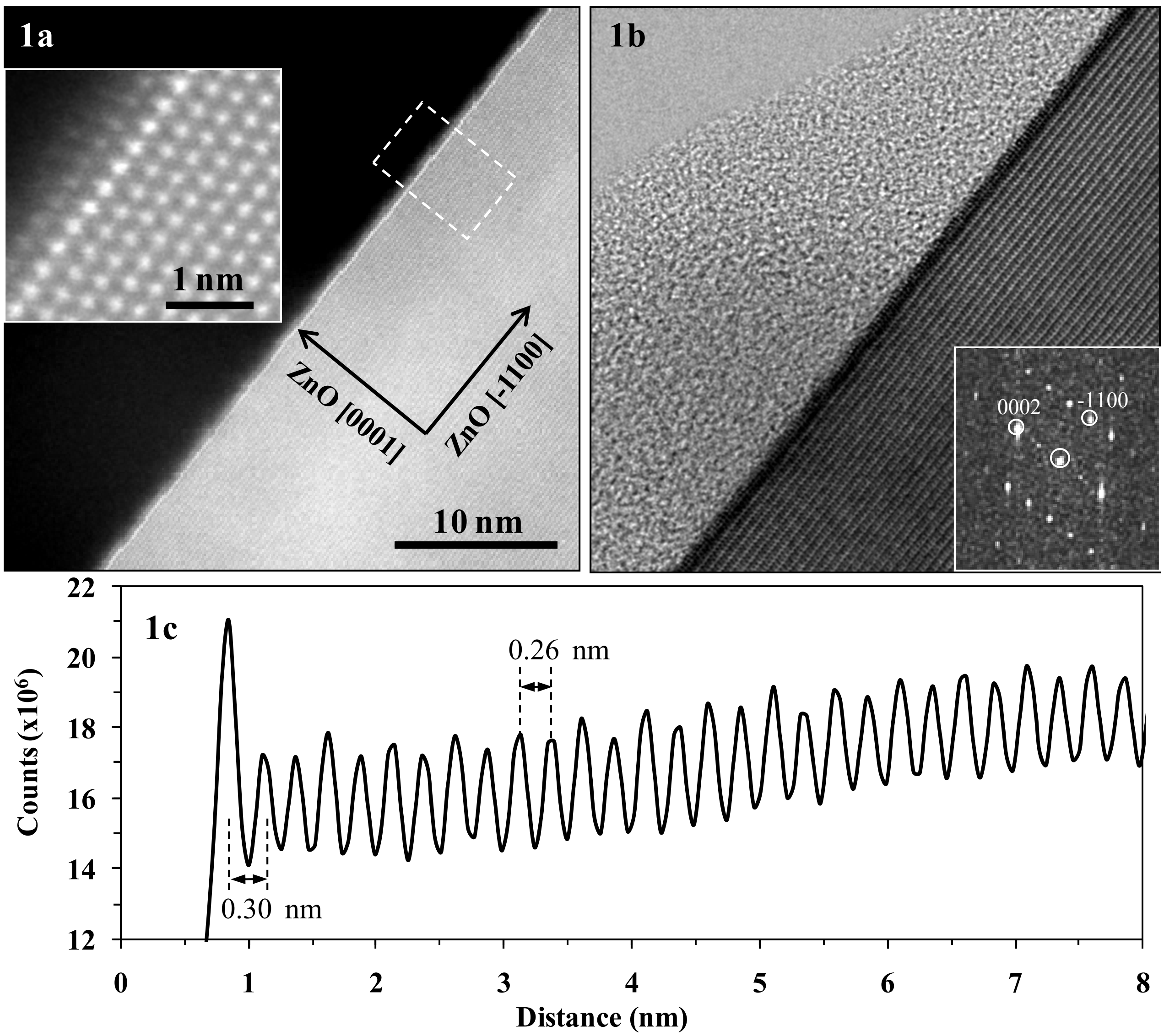

FIG. 1. a) HAADF image and b) the corresponding BF STEM image of a ZnO nanobelt. Magnified HAADF image is shown in inset of Fig. 1a and the computed diffractogram is shown in inset of Fig. 1b. An intensity profile across the ZnO (0001) lattice fringes within the rectangular box in Fig. 1a, projected along the ZnO [-1100] direction, is displayed in Fig. 1c.

https://doi.org/10.1017/S143192761005991X Published online by Cambridge University Press