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Low-voltage Electron-diffraction Microscopy Using SEM-based Microscope

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The roles of transmission electron microscopes (TEMs) and scanning electron microscopes (SEMs) with low acceleration voltages (in the range of several tens of kilovolts) have been converging. Owing to the growth of demand for light-element materials (i.e., radiation-sensitive materials) in green-innovation industries, the importance of analysis in this acceleration-voltage range has recently been increasing. Some projects have been developing aberration correctors for low-voltage TEMs and scanning transmission electron microscopes (STEMs) [1-4] and have reached atomic resolution. On the other hand, diffractive imaging, which provides a structural image of a specimen from a diffraction pattern with iterative phase retrieval, has started to open up the possibility of atomic resolution with an SEM-based microscope at low voltage [5, 6]. This imaging method has an advantage in terms of high resolution because resolution in this case is defined by recorded diffraction angle and lens aberration is not a critical factor. The developed microscope, which was used to verify atomic-resolution imaging of single-wall carbon nanotubes (SWCNTs) at 30 kV, is based on a conventional in-lens-type SEM (S-5500, Hitachi High-Technologies Corp.). The appearance of CNTs grown from a matrix is evaluated from SEM image obtained with this microscope, and certain parameters concerning a CNT (i.e., diameter, chirality) are evaluated from the obtained diffraction pattern. The results of these evaluations are used to characterize the crystallizability of the CNTs in low-damage range of acceleration voltage. An SEM with this diffractive-imaging function is presently being applied to SWCNTs, graphene, and nanoparticles. SEM has various functions for obtaining information concerning, for example, wide-area surface morphology, voltage contrast, and difference between elements and that between orientations of polycrystalline grains. Combining the functions for crystalline characterization from diffraction patterns and atomic-resolution structural analysis by diffractive imaging (collectively called "diffraction microscopy") with the inherent functions of SEM will increase the applications of electron microscopy in developing materials and devices (especially film, fiber, and particles). We would like to thank Prof. Shinohara's group at Nagoya University for growing the SWCNTs. Part of this work was supported by the Japan Science and Technology Agency (JST). [1] O. L. Krivanek et al., *Nature*, 464 571 (2010). [2] K. Suenaga et al., *Nat. Chem.*, 1 415 (2009). [3] U. Kaiser et al., *Ultramicroscopy*, 111 1239 (2011). [4] J. C. Meyer et al., *Nano. Lett.*, 8 3582 (2008). [5] O. Kamimura et al., *Ultramicroscopy*, 110 130 (2010). [6] O. Kamimura et al., *Appl. Phys. Lett.*, 98 174103 (2011).