Microsc. Microanal. 8 (Suppl. 2), 2002
DOI 10.1017.S1431927602105770

Development of A High Energy-resolution Soft-X-ray Spectrometer for A Transmission Electron Microscope

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A high energy-resolution soft-X-ray emission spectroscopy using a transmission electron microscope enable us to obtain the information of the density of states (DOS) of the valence band (occupied states) from identified small specimen areas. The DOS of the valence band together with the DOS of the conduction band (unoccupied states), which is obtained by electron energy-loss spectroscopy (EELS) [1], is imperative to understand the whole electronic structure.

We constructed a soft-X-ray spectrometer composed of a grating and a CCD detector [2,3]. B K-emission spectra of hexagonal boron nitride (h-BN) were obtained with an energy resolution of 0.6eV from a 1μm diameter specimen area. The spectra showed features similar to those of the reported B K-emission spectrum of h-BN obtained using a synchrotron orbital radiation facility. This spectrometer successfully showed that the DOS of the conduction band is obtained from specified small specimen areas by using a transmission electron microscope. Unfortunately, the spatial resolution of 1μm was not enough for nanometer scale analyses.

We have constructed a new soft-X-ray spectrometer with better performance (the special resolution, the energy resolution and the measurable energy range) than our previous spectrometer. Figure 1 shows a schematic diagram of the improved soft-X-ray spectrometer. The size of the CCD detector is 27.6x27.6mm^2 (2048x2048 channels). The collection angle of the present spectrometer is 6.5x10^{-4} sr, which is 3.5 times larger than that of our previous spectrometer. The larger collection angle enables us to use a smaller electron probe of 400nm in diameter. Energy resolution is improved from 0.6eV to 0.4eV at B K-emission energy due to a smaller pixel size of the CCD. The new spectrometer chamber can set three gratings. Energy range from 60eV to 1200eV is accessible by using two different grating.

Figure 2 shows B K-emission spectra of h-BN and cubic BN (c-BN). Each spectrum was obtained from single crystalline areas of 400nm in diameter with a detection time of 1 hour. Each spectrum shows one large peak indicated by an arrow. It should be noted that the energy of the peak of h-BN is smaller than that of c-BN. This result is consistent with the theoretically calculated DOS of h-BN and c-BN [4]. Those peak intensities are assigned to σ-bonding states between B and N atoms. The σ-bonding length of h-BN is shorter than that of c-BN. It causes a

larger energy difference between bonding states and anti-bonding states in h-BN than that in c-BN. Thus, the difference of peak positions observed in Fig.2 can be assigned to the different B-N distances between h-BN and c-BN.

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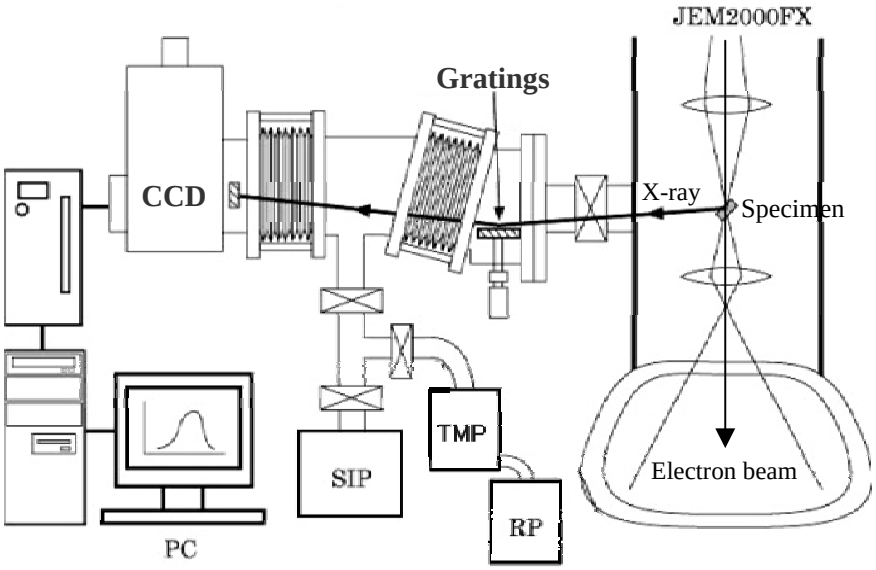

FIG.1 Schematic diagram of the improved soft-X-ray spectrometer attached to a TEM using a grating and a CD detector.

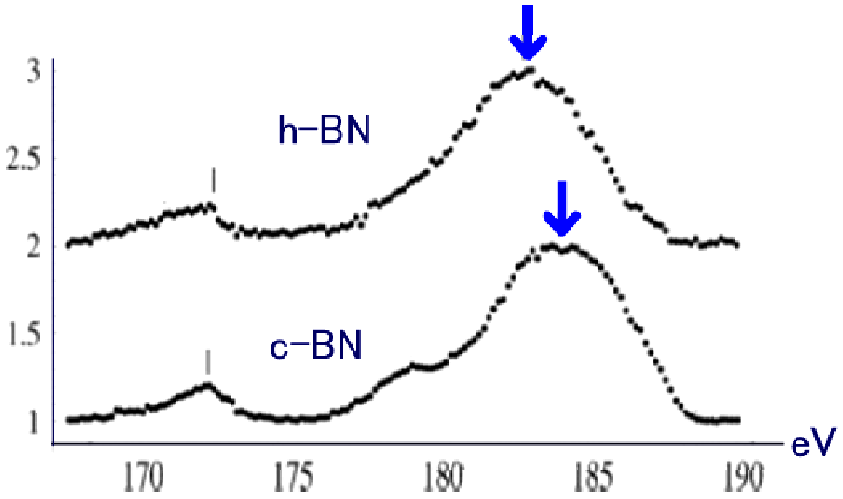

FIG.2 B K-emission spectrum of hexagonal boron nitride and cubic boron nitride obtained from 400nm diameter specimen areas with an energy resolution of 0.4 eV.

https://doi.org/10.1017/S1431927602105770 Published online by Cambridge University Press