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# Development of wavelength-dispersive X-ray spectrometer for a conventional analytical transmission electron microscope

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Characterization with nm-scale spatial resolution is important for developments of new functional materials. Recently, Terauchi et al. [1-3] constructed a compact wavelength-dispersive X-ray spectrometer, referred as the original spectrometer, for a transmission electron microscope (TEM) and succeeded to obtain a density of states (DOS) of the valence band with an energy resolution of better than 1 eV by using a TEM. It implies that the DOS of the valence band (by X-ray emission spectroscopy: XES) and the conduction band (by electron energy-loss spectroscopy: EELS) can be obtainable from a specified small specimen area based on a transmission electron microscopy [4]. Furthermore, the spectrometer dose not disturb the spatial resolution of a TEM, because it does not have a moving mechanism as in a case of WDS spectrometers of EPMA.

We have started a project to renew the WDS spectrometer to attach to a conventional analytical TEM. Basic concept of the first spectrometer (high energy-dispersion version) is the same as that of the original one. It composed of 1) three varied-line-spacing (VLS) gratings, 2) a back-illumination type CCD detector and 3) X-ray collection mirrors. The measurable energy range is from 60 to 1200 eV. Compared to the original spectrometer, energy dispersion in an energy range of 60-400eV was improved by about two times.

This first spectrometer is different from the original one in the take off angle of X-ray to measure. The original one was designed to measure X-rays emitted in almost horizontal direction. As a result, a specimen always has to be tilted more than 20 deg. for measurements. It is not convenient for a conventional analytical TEM. Thus, this new instrument is designed to measure the X-ray in the direction with an angle of elevation of about 35 deg. A specimen tilt for XES measurements is no more needed. The spectrometer is firstly installed in a TEM of JEM2010 as shown in FIG.1. A silicon L-emission (valence bands→$L_{2,3}$-shell) spectrum obtained by the first spectrometer is shown in FIG.2. The spectrum represents the DOS of the valence bands with s-symmetry showing peak structures labeled by symmetry characters of L2', L1, X4 and L3'.

The second WDS spectrometer is designed to cover an energy range of 60-1200 eV by using newly designed two VLS gratings. The energy dispersion of the spectrometer is designed smaller than that of the first spectrometer to obtain a larger collection angle, because of an extremely small emission efficiency of the soft-X-ray region to measure. Figure 3 shows a prototype of the second spectrometer attached to the same TEM shown in FIG.1. The spectrometer is equipped an automatic exchange mechanism of the two gratings for easy operation. X-ray collection mirrors are newly designed for the spectrometer. When this spectrometer is installed in a high energy-resolution EELS microscope, the microscope can get the valence and the conduction band DOS from a specified small specimen area.

This project is conducted under a name of "Development of an EELS/XES electron microscope for electronic structure analyses", which is one of the leading projects of Ministry of Education, Culture, Sports, Science and Technology, Japan.

References: [1] M. Terauchi et al.: J. Electron Microscopy, **50** (2001) 101. [2] M. Terauchi and M. Kawana: Microsc. Microanal., **8** suppl.2 (2002) 644. [3] M. Terauchi and M. Koike: Microsc. Microanal., **9** suppl.2 (2003) 894. [4] M. Terauchi, Micro. Res. Tech., (2006) in press.

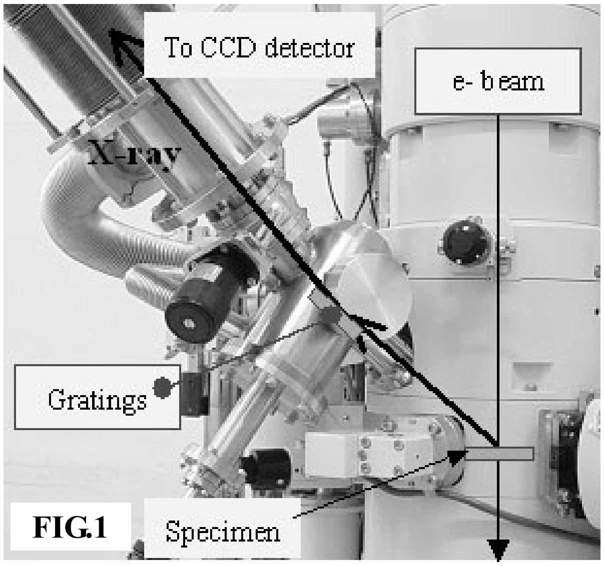

**FIG.1**

FIG.1
First WDS spectrometer (high-dispersion type) attached to a conventional TEM of JEM2010. The spectrometer composed of three gratings and a CCD detector.

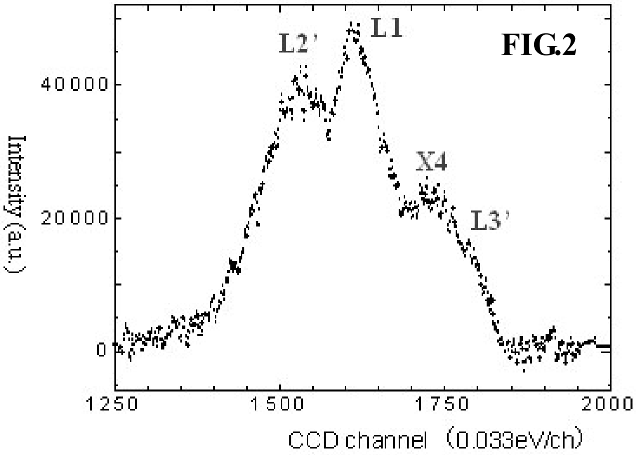

**FIG.2**

FIG.2
Si L-emission spectrum of Si single crystal obtained by the first spectrometer in FIG.1. The spectrum shows peak structures which can be assigned to special symmetry points of the electronic band structure of the silicon.

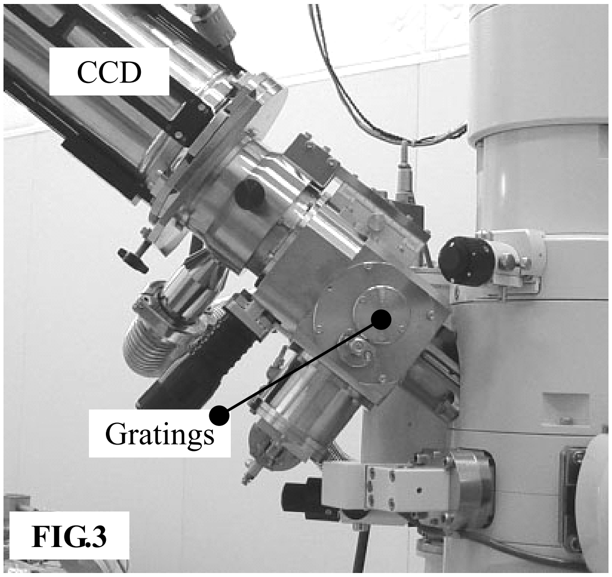

**FIG.3**

FIG.3
Second WDS spectrometer (prototype for commercial purpose) attached to the same TEM shown in FIG.1.