Development of Large Solid Angle SDD for TEM and its Applications


* JEOL Ltd., 3-1-2 Musashino, Akishima, Tokyo 196-8558, JAPAN
** Kyushu University, 744 Motooka, Nishi-ku, Fukuoka, Fukuoka 819-0395, JAPAN

An Energy Dispersive X-ray Spectrometer (EDS) has been one of the most important devices for analytical Transmission Electron Microscopy (TEM), since it can perform simultaneous multi-elemental analysis on a sample having wide range of thickness and atomic numbers. However, an EDS detector can detect just a few percentages of X-ray from a sample, because its size is as small as 50 mm² so far and there are obstacles near the specimen such as an objective lens pole-pieces, an anti-contamination device and a sample holder. Therefore, TEM-EDS analysis needs relatively long acquisition time and the samples sometimes suffer contamination and electron irradiation damage.

We have developed a new large solid angle EDS detection system for TEM. The system includes a silicon-drift-type detector (SDD) with large sensing area of ~100 mm². Figure 1 shows an appearance of the new EDS detector. The detector is a side-entry-type as same as Si (Li) EDS detectors. Therefore, one can easily make a swap with an old or damaged detector. The new detector has automatic retractable mechanism to protect the sensor from the high-energy backscattered electron irradiation, which occurs on LOWMAG mode, where no magnetic field is applied on a sample. The sensor is isolated from a microscope column with an ultra-thin polymer window, to block adsorption of sublimated gases from a specimen on the cooled sensor surface. The window has 100 % open area with no supporting grid. Thus, the sensor can receive the X-ray from a sample without absorption with the support grid.

Figures 2 and 3 show EDS spectra from a Ni evaporation film and carbon film, obtained by a 200 kV microscope (JEM-2100) equipped with the new detector. The ratio of Ni Lα to Ni Kα is approximately 1.1, suggesting high sensitivity of low-energy X-ray. The ultimate energy resolutions of the new detector, measured so far, are <130 eV in FWHM at Mn Kα and <50 eV at C Kα (Fig. 3).

Because of the large sensor area of ~100 mm² and the shorter distance between the sensor and the specimen realized by well-considered design, the new detector provides much larger solid angles. Those in high-resolution and ultrahigh-resolution configurations are 0.98 sr and 0.80 sr respectively. Therefore, an X-ray mapping can be performed with much shorter acquisition time. Figure 4 shows the results of X-ray mapping sized 256 x 256 pixels for the grain boundary in a SIALON, obtained by JEM-ARM200F equipped with the new detector. The total X-ray acquisition time was just 40 seconds. Even with the acquisition time less than 1 minute, trace elements such as Y, O, and Al in the grain boundary were detected clearly, suggesting higher detection sensitivity compared with old EDS detectors. Moreover, the detector can acquire atomic resolution X-ray mapping in combination with Cs-corrected TEM/STEM machines such as ARM200F. Figure 5 shows the result of atomic resolution X-ray mapping sized 128 x 128 pixels for SrTiO₃<100>. The total X-ray acquisition time was about 10 minutes. The probe current was about 120 pA. The atomic columns of Sr and Ti were clearly observed. Figure 6 shows the result of atomic resolution X-ray mapping sized 64 x 64 pixels for GaAs<110>. The probe current was about 120 pA. The total of X-ray acquisition time was about 14 minutes. Although the atomic distance of Ga-As dumbbell is
as short as 0.14 nm, the result clearly distinguished the dumbbell.

As mentioned above, our new EDS system, incorporated with the 100-mm²-sized sensor, has significantly high X-ray collection ability. Therefore, it can provide shorter acquisition time and higher throughput compared with old Si (Li) EDS detectors.

Fig. 1: Appearance of the new EDS detector for TEM.

Fig. 2: EDS spectrum from a Ni evaporated film, obtained with JEM-2100.

Fig. 3: EDS spectrum from a carbon film, obtained with JEM-2100.

Fig. 4: Elemental map for the grain boundary in a SIALON, obtained with JEM-ARM200F.

Fig. 5: Elemental map for SrTiO₃<100>, obtained with JEM-ARM200F.

Fig. 6: Elemental map for GaAs<110>, obtained with JEM-ARM200F.