5–10 Challenge for Nanoscale Analysis by X-ray Spectroscopy in Combination with Electron Microscopy

— Development of a Multilayer Grating Spectrometer for Use with Electron Microscopes —

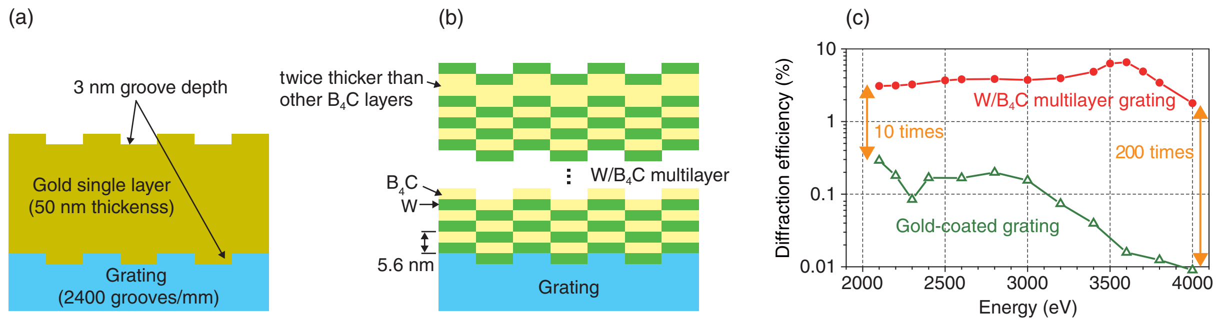

Fig.5-28 Schematics of (a) a gold-coated grating and (b) an aperiodic W/B_4C multilayer grating. (c) The measured diffraction efficiencies of both gratings are plotted as functions of photon energy at a constant angle of incidence
The W/B_4C multilayer grating shows uniformly high diffraction efficiency across the measured energy range.

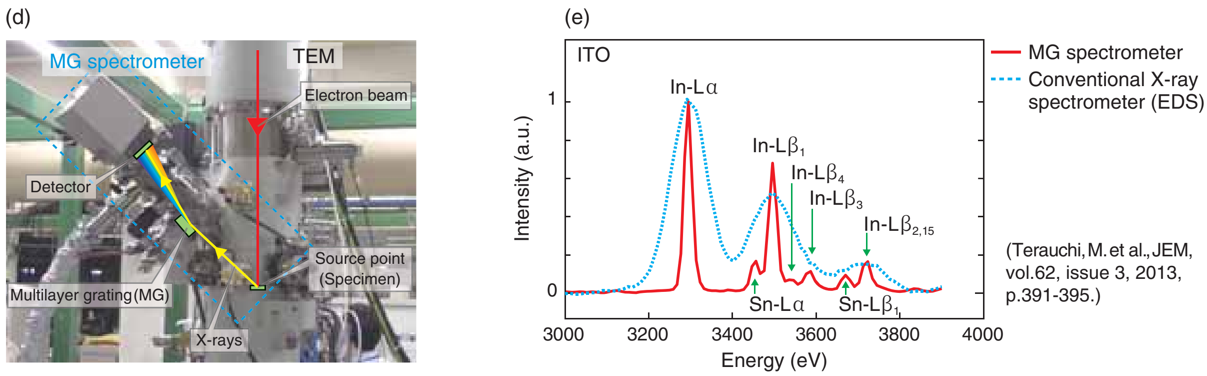

Fig.5-29 MG spectrometer installed in (d) a TEM and (e) emission spectra from ITO
The MG spectrometer better resolves the fine structure of the spectra, such as the Sn-Lα and In-Lβ_1 peaks, than conventional EDS.

Electron microscopes (EMs) are useful tools for the structural analysis of materials at nanometer scales. X-rays are generated when a material is irradiated by an electron beam. If the X-ray intensity distribution is plotted as a function of the photon energy by using an X-ray spectrometer with a diffraction grating featuring many narrow grooves on its surface, we can analyze the valence electronic structure underlying the material properties. As conventional soft X-ray gratings are coated with a thin gold film, their use becomes impractical at energies around the gold absorption edges (approximately 2.2 keV). To overcome this problem, we designed an aperiodic W/B_4C multilayer structure and applied it to a wideband multilayer grating covering 2.0–4.0 keV at a constant angle of incidence.

Fig.5-28 presents schematics of an Au-coated grating (AG) (a) and the designed aperiodic W/B_4C multilayer grating (MG) (b). The measured diffraction efficiency curves of both gratings are plotted in panel (c). The diffraction efficiencies of the MG at 2.1 keV and 4.0 keV are 10 and 200 times higher than those of the AG, respectively. The MG also shows uniformly high efficiency over the entire energy range. This result is attributable to the reflective characteristic of the B_4C layer just below the topmost W layer, whose thickness is twice that of the other layers in the structure, as shown in Fig.5-28(b).

Fig.5-29 is a photograph of the MG spectrometer installed in a transmission EM. Also shown are the emission spectra from indium-tin-oxide measured by the MG spectrometer and a conventional energy-dispersive spectrometer (EDS). The L emission spectra of Sn and In (e.g., Sn-Lα and In-Lβ_1) are more clearly resolved by the high-resolution MG spectrometer than by EDS.

By combining X-ray spectroscopy with EM, we have established a unique technique for extracting morphological and electronic structure information. This technique is expected to assist the development of electronic devices and functional materials.

The present study was partly supported by the project of Collaborative Development of Innovative SEEDs from the Japan Science and Technology Agency (JST).

Reference

Imazono, T., Development of a Soft X-ray Flat-Field Spectrograph in the 50-4000 eV Range and Its Application to Electron Microscopes, Oyo Butsuri, vol.83, no.4, 2014, p.288-292 (in Japanese).