

## Construction of a SXES Spectrometer for a Conventional SEM

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We have been developed and tested soft-X-ray emission spectroscopy (SXES) instruments by attaching to TEM and EPMA. The spectrometers have an energy range from 50-4000eV by using four varied-line-spacing (aberration corrected) gratings [1,2,3]. This SXES spectrometer informs us energy states of valence electrons (bonding electrons) from specified specimen areas in electron microscopy, which is hardly obtained by EELS and EDS. Since there are many atomic resonances in soft-X-ray energy region, this approach can be used not only as a probing method to evaluate the energy states of valence electrons but also a sensitive tool for elemental and chemical identifications. Thus, various applications can be expected through this approach. The spectrometer can also be retrofitted to a conventional SEM, but had not been tested. Here, we present development and initial results of the SXES spectrometer mounted on a conventional SEM.

Figure 1 shows a photo of the newly manufactured SXES spectrometer attached to a conventional SEM (JSM-6480LV), which has a low-vacuum mode of 10-270 Pa. This spectrometer has the same optical design as that applied for TEM [1]. X-ray collection mirrors are newly designed to achieve a larger collection angle. For signal detection, a multi-channel plate (MCP) optically coupled with a conventional CCD camera was used. This detector (MCP+CCD) is operated in photon-counting mode, in which the CCD has to be set in a short exposure time resulting in a no-effect of dark-current without cooling. An additional pumping system, which is a combination of a turbo-molecular pump and a rotary pump, was added to maintain the vacuum condition around the MCP in high enough.

Figure 2 shows an Al-L emission spectrum obtained with a probe current of 10-20 nA and a detection time of 3 min. Since a SEM can be operated with higher probe currents and excitation volumes of specimen in a SEM is much larger, the detection time can be minimized to one order or less than that of a TEM. At the right hand end of the intensity distribution, L<sub>2</sub> and L<sub>3</sub> edges are seen. The energy resolution evaluated at the L<sub>3</sub> edge is 0.2 eV.

Figure 3 shows a 2<sup>nd</sup>-order spectral intensity of B-K emission from powder specimens of LaB<sub>6</sub> and amorphous boron (am-B) acquired with a probe current of 10-20 nA and a detection time of 5 min. The spectrum of LaB<sub>6</sub> also shows a 1<sup>st</sup>-order spectrum intensity of La 4d-5p. Signal-to-noise ratios are not sufficient, but there can be seen an apparent difference in the right-hand side end of the B-K peaks between LaB<sub>6</sub> and am-B, where corresponds to the top of the valence bands. The spectrum of LaB<sub>6</sub> shows an apparent feature corresponds to Fermi edge, showing different chemical states of boron in this material.

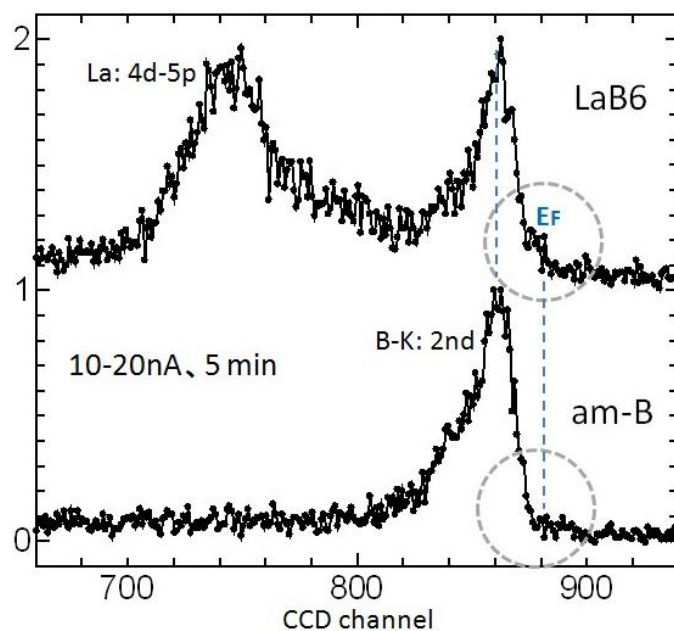
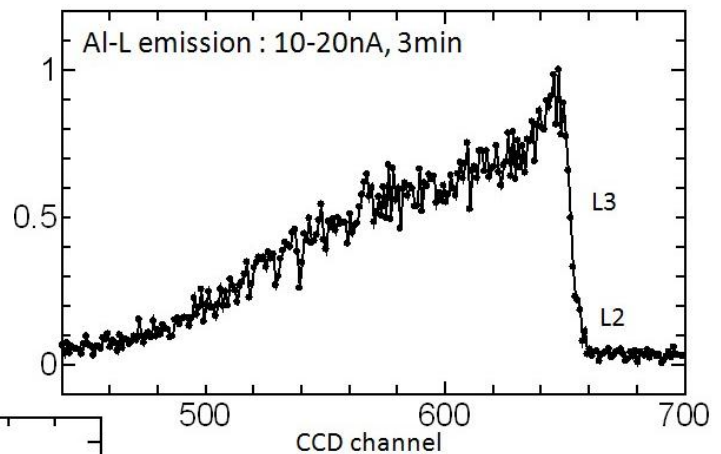
## References

- [1] M Terauchi *et al.*, Journal of Electron Microscopy, **61** (2012) 1.  
 [2] T Imazono *et al.*, Applied Optics, **51** (2012) 2351.  
 [3] M Terauchi *et al.*, Microscopy 2013; doi: 10.1093/jmicro/dfs129.



**Figure 1.** Photo of the newly manufactured SXES spectrometer attached to a conventional SEM (JSM-6480LV). As a detector, a multi-channel plate optically coupled with a conventional CCD camera was used.

**Figure 2.** Al-L emission spectrum obtained with a probe current of 10-20 nA and a detection time of 3 min. At the right hand end of the intensity distribution, L<sub>2</sub> and L<sub>3</sub> edged are seen. The energy resolution evaluated at L<sub>3</sub> edge is 0.2 eV.



**Figure 3.** Spectral intensities of 2<sup>nd</sup>-order peak of B-K emissions from powder specimens of LaB<sub>6</sub> and amorphous boron (am-B). The spectrum of LaB<sub>6</sub> also shows a 1<sup>st</sup>-order peak of La 4d-5p. There can be seen an apparent difference in the right-hand side end of B-K emission intensities, related to the top of the valence bands. The spectrum of LaB<sub>6</sub> shows apparent feature corresponds to Fermi edge.