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Chemical States Analysis of Trace-boron by using an Improved SEM-SXES

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X-rays originate form electronic transitions from valence bands (bonding electron states) to inner-shell electron levels inform us elementally specific chemical states in materials. The energies of those X-rays range from about 0.1 to a few keV, soft X-ray region. Thus, soft X-ray emission spectroscopy (SXES) based on electron microscopy (EM) can be a sensitive tool for elemental and chemical identifications of specified specimen areas. For that purpose, a grating spectrometer for SXES-EM has been realized as a commercial instrument with an energy resolution of 0.2-0.3 eV at Al-L emission (73 eV) [1,2].

A test SXES-SEM instrument with a micro-channel plate (MCP) detector combined with a CMOS camera has been applied for state analyses of bulk specimens [3]. It performed an energy resolution of 0.13 eV at Al-L emission. To improve the energy resolution of the system, the MCP with a channel pitch of 15 μm has changed to a new one with that of 7.5 μm. Optical component between the MCP and the CMOS camera was readjusted in its magnification. Figure 1 shows Al L-emission spectra of a bulk specimen obtained by the improved system in photon counting mode. A small step of L_2-edge (E_F to L_2-shell) and a large L_3-edge (E_F to L_2-shell) is clearly resolved. The energy resolution evaluated from the L_3-edge by taking into account the thermal broadening of Fermi distribution function is 0.08 eV, which is better than that of the previous one. Although the energy resolution has improved, it takes a longer acquisition time to have a good signal to noise ratio in photon counting mode compared to that of analog integration mode. Thus, analog integration mode has adopted for the present trace-boron analysis, unfortunately the energy resolution is a few times lower than that of photon counting mode.

Figure 2 shows an SEM image of a carbon steel specimen. Average contents of B, P, and C are 45, 53, and 41 ppm (wt.%), respectively. The image shows several precipitates with different sizes and shapes are distributed in the matrix. The SXES spectra obtained from the numbered areas in figure 2 are shown in figure 3. Accelerating voltage, probe current and acquisition time were 5 kV, 170 nA and 5 min, respectively. The spectrum of area 3 (matrix area without any precipitates) shows mainly C-K and O-K signals, which may originate from contamination and oxidation of the specimen surface, respectively. It should be noted that there is no apparent B-K intensity in the spectrum. On the other hand, spectra obtained from precipitate areas show characteristic peaks and show different features each other. Area 1 shows mainly C-K and a little B-K. Area 2 shows a dominant B-K and smaller N-K and S-L. Area 4 shows B-K and S-L. Area 5 shows a dominant B-K and small N-K and Si-L. It can be noticed that spectra with dominant B-K intensity, 2 and 5, also show apparent N-K and O-K intensity. O-K intensity of those spectra is larger than that of area 3, matrix without any precipitate. Those results suggest a presence of boron nitride and boron oxide in 2 and 5 areas. A much lower content of B (11 ppm) specimen also includes precipitates and successfully obtained B-K intensity form some precipitates. To improve the detection efficiency of the SXES spectrometer, a now grating is now under preparation.

References:
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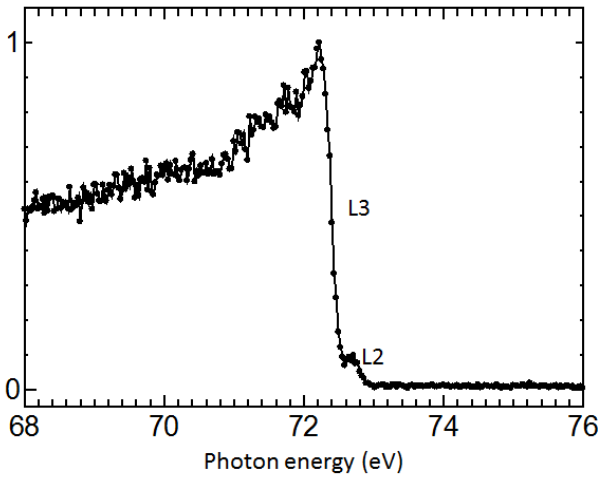

Figure 1. Al L-emission spectrum with an energy resolution of 0.08 eV obtained by photon counting mode.

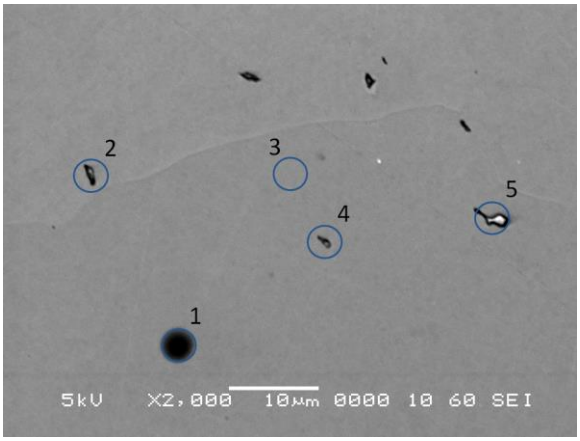

Figure 2. SEM image of a carbon steel specimen with minor elements of B (45 ppm), P (53 ppm) and C (41 ppm).

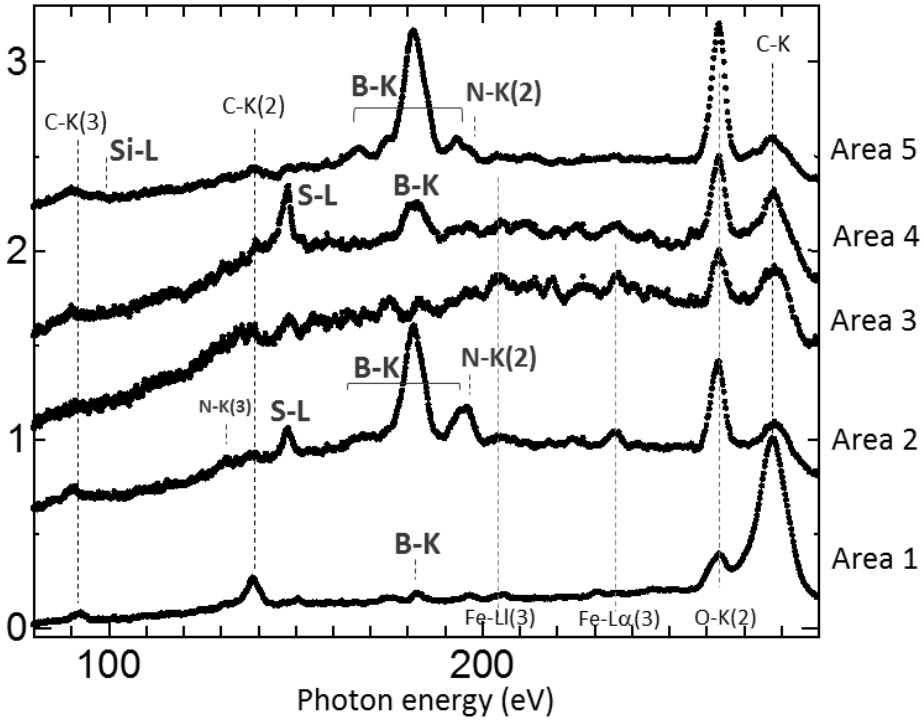

Figure 3. SXES spectra obtained from areas 1-5 shown in figure 2 by analog integration mode. Precipitates show a variety in compositions. Spectra of 2 and 5 with dominant B-K intensity also show N-K and larger O-K intensity than area 3 (matrix), suggesting a presence of B-N and B-O compounds.

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