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Chemical State Mapping via Soft X-rays using a Wavelength Dispersive Soft X-ray Emission Spectrometer with High Energy Resolution

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A new wavelength dispersive soft X-ray emission spectrometer (WD-SXES) consisting of X-ray reflection mirrors, newly developed diffraction gratings, and a detector of a charged-couple device (CCD) has been developed for soft X-ray emission spectroscopy [1, 2, 3]. The WD-SXES with two types of diffraction gratings, JS50XL and JS200N, nominally covering an energy range between 50 and 210 eV and with a high sensitive back-thin-type CCD as the detector has successfully been installed to electron probe X-ray microanalyzers (EPMAs) for commercial use [4]. The energy resolution of this WD-SXES is nominally 0.3 eV, which is one order of magnitude better than that of conventional WDSs with layered dispersion elements used for detecting light elements in EPMAs. One other useful characteristic of this WD-SXES is parallel detection of the X-rays, which makes possible to operate just like conventional energy dispersive spectrometers for EPMAs. In this presentation, chemical state mapping is reported by means of soft X-rays using this high energy resolution WD-SXES.

In this study a JXA-8100 EPMA with the WD-SXES was used for chemical state mapping of an Al-B alloy as shown in Fig. 1. A backscattered electron image of the alloy is shown in Fig. 2. It mainly consists of two phases; one with brighter and the other with darker contrast. The former is aluminum matrix and the latter is Al_2B. To acquire the emission spectral mapping the following conditions were used: the accelerating voltage of 7kV; the beam size of about 1 μm; the beam current of 0.1 μA; the X-ray emission spectral range of 55 and 215 eV dispersed by JS200N grating; the pixel number of specimen of 120 × 120 pixels; the scan step interval in the stage movement mode of 0.5 μm; and the acquiring time per pixel of 30 s. Four peaks corresponding to Al-L, B-K (n = 1 and 2) and C-K (n = 2) are clearly observed with high energy resolution. Here, n denotes the spectral order. Two spectra extracted from the 3 × 3 pixels of aluminum matrix and of Al_2B inclusion are shown in the energy range between 55 and 85 eV in Fig. 4. In each spectrum the Al-L emission is observed with a sharp edge in the higher energy side. The edge of the aluminum matrix corresponds to the Fermi energy of aluminum metal. It should be noted that the corresponding edge of Al_2B is shifted to higher energy side by about 1 eV. Using the acquired and stored spectrum data, chemical state mapping was performed using a reconstruction program. The energy range of ROI-1 (region of interest) was selected from 72 to 73.5 eV whereas that of ROI-2 was from 73.5 to 75 eV as shown in Fig. 4. In the former range is included the Al-L emission spectrum peak of metallic aluminum excluding that of Al_2B, whereas in the latter the situation is the other way round. The resulting maps are shown in Fig. 5. The contrast in the ROI-1 map is reversed in the ROI-2 map as expected. Note that the difference between two ROIs is only 1.5 eV. The study confirms that the high energy resolution performance of the WD-SXES system is useful to map out difference in the chemical state. In addition, other characteristic soft

X-ray lines such as Li-K (54 eV), Be-K (109 eV), B-K (183 eV), Mg-L (50 eV), Al-L (73 eV), Si-L (91 eV), P-L (119 eV), S-L (149 eV) and Cl-L (184 eV) are located in the energy range covered with the present WD-SXES system. The detectable energy range can be extended to the K emission spectra of C, N, O and F for the use of higher order X-ray spectra. The WD-SXES will certainly be used in the wide field for the characterization of various materials, especially of advanced materials.

References

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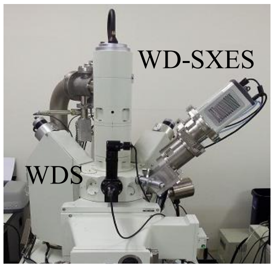

Fig.1 Newly developed WD-SXES installed to an EPMA of JEOL JXA-8100

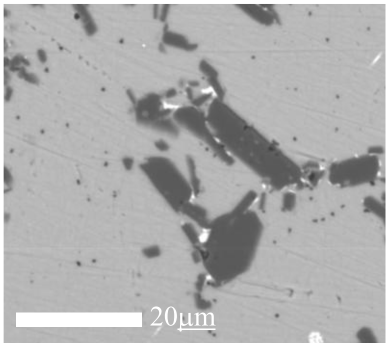

Fig. 2 A backscattered electron image of Al-B alloy

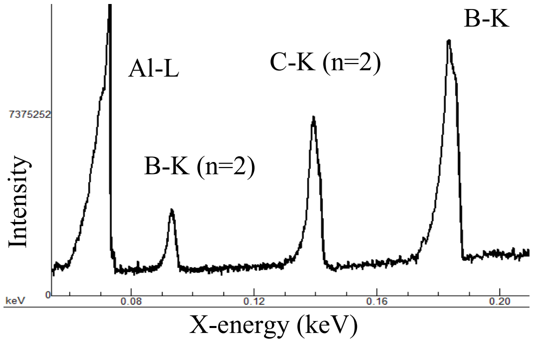

Fig. 3 Cumulative spectrum from all the pixels by spectrum map

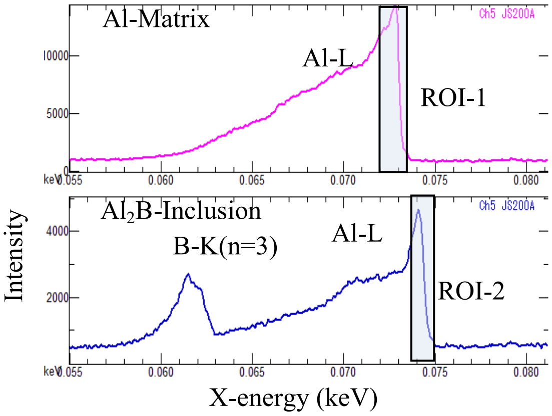

Fig. 4 Comparison of Al-L emission spectra between Al matrix and Al$_2$B inclusion in Al-B alloy. ROI-1 (72-73.5 eV) and ROI-2 (73.5- 75 eV) were selected.

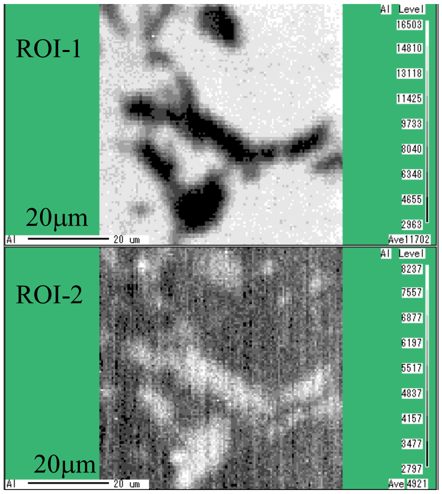

Fig. 5 Comparison of Al-L chemical state maps between Al matrix (ROI-1; 72-73.5 eV) and Al$_2$B inclusion (ROI-2; and Al matrix73.5 – 75 eV).