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Elemental Analyses of Heat Resistant Steels by High-Energy Resolution EDS Analyzer Based on Superconducting-Tunnel-Junction Array

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The creep strength and creep life of heat-resistant steels can be improved by adding small amounts of light elements such as B, C, and N. For example, nanosized (<100 nm) inclusions of light element compounds such as MX carbonitrides (M: Nb, V, Cr; X: C, N) increases the creep strength of 9% Cr steels [1]. Thus, to understand the effects of the light elements in the heat-resistant steels and to improve those mechanical properties, it is very important to evaluate amounts of each light element and their spatial distribution in there, for example, by carrying out qualitative elemental mappings of the light elements.

An energy-dispersive X-ray spectroscopy (EDS) analyser combined with a scanning electron microscope (SEM) is suitable to obtain spatial and quantitative information on the elemental composition of a sample non-destructively. In particular, low-acceleration-voltage SEMs (LVSEMs) theoretically allow evaluating those informations of a sample surface with a nanometer lateral resolution [2]. However, it isn't suitable to use energy-dispersive X-ray detectors such as silicon drift detectors (SDDs) or Si(Li) detectors in the conventional EDS analysers for obtaining X-ray spectra from samples in LVSEMs, because emitted X-ray from samples in LVSEMs are only soft X-ray and the energy-resolving power of conventional energy-dispersive X-ray detectors is insufficient to clearly resolve such soft X-rays. For example, in case of the elemental analyses to the 9% Cr steels, the conventional EDS analysers cannot resolve the Cr-$L\alpha$ line (500 eV) and the O-$K\alpha$ line (525 eV) into two individual peaks [3].

In contrast, energy-dispersive X-ray detectors based on arrays of superconducting-tunnel-junctions (STJs) have simultaneously exhibited excellent energy resolution of <10 eV, relatively large detection area of >1 mm^2, and high counting rate capability of >500 kcps for soft X-rays less than 1 keV [4,5]. We have developed the SEM utilizing STJ array as an EDS system, which is abbreviate as SC-SEM hereafter, in order to analyse light element dopants in structural or functional materials [6]. In this work, we performed elemental analyses of the 9% Cr steel in order to demonstrate performance of the SC-SEM at low-acceleration-voltage.

Figure 1 shows a cross-sectional schematic illustration of the SC-SEM. The SC-SEM consisted of an SEM with a tungsten filament and the STJ array detector. The STJ array was cooled to 0.31 K on a cold stage of a cryogen-free helium-3 cryostat. X-rays emitted from the sample by the electron beam were detected by the STJ array via the polycapillary collimating X-ray lens and two X-ray windows. In order to improve the collection efficiency, a polycapillary collimating X-ray lens was installed in the system. The collection efficiency of the SC-SEM was 0.34 msr, which was about 1/100 times smaller than that of the SDDs [7].

Figure 2(a) shows a SEM image of the 9% Cr steel. The acceleration voltage and probe current of the SEM were 5 kV and about 10 nA, respectively. The black and white inclusions of sub-micrometer size can be seen from the SEM image. To evaluate the elemental composition of the black inclusion, X-ray spectra of point 1 and 2 in the SEM image were measured by using the SC-SEM. The spectra were displayed in figure 2(b). The measurement time was 3600 s. In the spectrum of point 1, Fe-*L*, Cr-*L*, and O-*K* peaks were observed, and Cr-*L*l (500 eV) and O-*K*α (525 eV) peaks can be clearly separated. A full width at half-maximum value for the peaks was about 10 eV, which was about 5 times higher than that of the SDDs. In contrast, in the spectrum of point 2, the N-*K*α peak was observed in addition to peaks observed in the spectrum of point 1, and the intensity of O-*K*α peak was about 3 times higher than that in the spectrum of point 1. As a result of comparison with the two spectra, it is supposed that the black inclusion is the steel containing a lot of oxygen and a little nitrogen [8].

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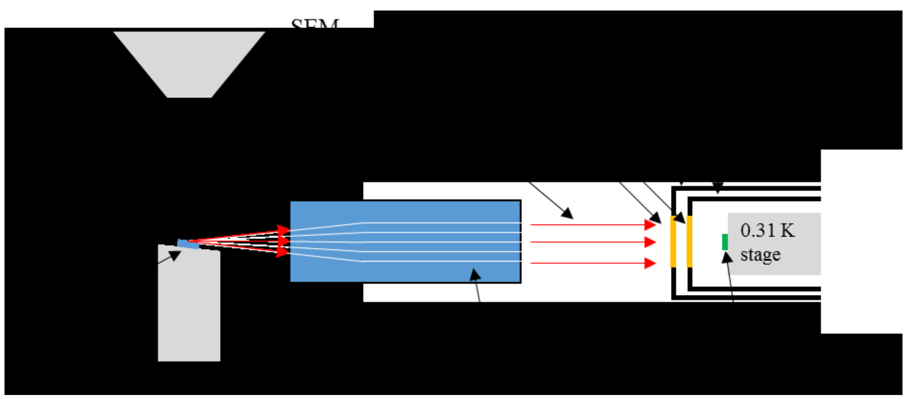

Figure 1. Cross-sectional schematic illustration of the SC-SEM.

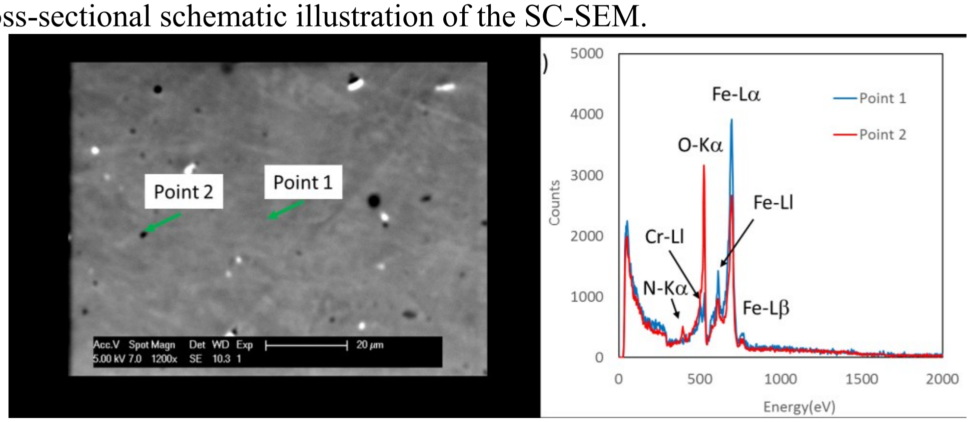

Figure 2. (a) SEM image and (b) X-ray spectra for the 9% Cr steel.

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