Characterization of multi-phase steel using collection-angle controlled BSE images

Sato K. ¹, Sueyoshi H. ², Yamada K. ²

¹JFE Steel Research Laboratory, Chiba, Japan, ²JFE Steel Research Laboratory, Fukuyama, Japan

Email of the presenting author: ka-sato@jfe-steel.co.jp

Microstructural characterization is crucial for designing advanced steels. Cs-corrected STEM has been successfully applied to the studies of nanometer-sized precipitates and interfaces at sub-nanometer resolution. For the quantification of particle size and precipitation ratio of alloying elements, particle beams such as synchrotron radiation and neutron have been extensively used.

Compared to the abovementioned methods, SEM has been regarded as a supporting technique. However, improvements in both spatial resolution at low-voltages and multi imaging detector design are giving rich information on the “real” surfaces.

For optimizing the microstructure of steels, characterization using SEM is powerful because it allows both low and high magnification observation. SEM specimens are often etched in order to differentiate the different phases as topographic information in steels. This is an “indirect” method of characterization, which does not give precise structural information. Consequently, we have been searching for a more direct imaging technique. Aoyama et al have done a systematic measurement of oxide on steel by changing the acceptance angle of back-scattered electron (BSE) images¹. Low angle (angle θ is measured from the surface) BSE images exhibit strong channeling contrast, whereas high angle BSE gives atomic number contrast. As shown in Fig.1a, sub-micron precipitates exhibit much higher visibility when low angle BSE is collected. The poor contrast in Fig.1b recorded at larger detection angles is not due to degraded probe size but due to the contribution of BSEs from larger volume. As can be seen in Fig.1d, the secondary electron image obtained at the same working distance as Fig 1b shows little image degradation.

We have found a new technique of selective imaging of martensite (M) phase in a ferrite (F)-M dual phase steel. BSE images at 10-15 kV were recorded by systematically changing θ. When θ was 30-45°, strong channeling contrast was observed. Under this θ, it is the low energy-loss electrons that mainly contribute to the contrast. As θ increases, M phase exhibits a high contrast. When θ exceeds 60°, a selective imaging of M phase was attained. This is not because martensite has a larger mean atomic number than ferrite, but is due to the fact that martensite has a high dislocation density. This is consistent with the fact that martensite always exhibits dark contrast in TEM bright field images regardless of the crystal orientation. Low angle BSE will allow high resolution characterization of lath structure and small precipitates, while high angle BSE gives quantitative measurement of the volume fraction and distribution of the second phase.

Fig. 1: Nanometer-sized carbide in a high strength steel observed at 15 kV. BSE images a) and b) and SE images c) and d) were recorded at two working distances 2 mm (a and c) and 20 mm (b and d). A higher visibility of carbide was achieved for Fig. 1a where collection angles were between 31°-45° than 1b whose collection angles were 77°-81°.

Fig. 2: Selective imaging of martensite (M) in dual-phase steel. BSE images were recorded at 15 kV. Figs 2a and 2c were taken with the collection angles of $\theta=31^\circ$-$45^\circ$ whilst figs 2b and 2d were taken at $\theta=63^\circ$-$70^\circ$. Lath structure is clearly seen at low $\theta$, while selective imaging of martensite was attained at high $\theta$. 