SEM-TEM combined characterization of precipitates and second phase

Sato, K.[1]

[1] JFE Techno-Research, Japan

Multiscale analyses ranging from mm, sub-μm to sub-nanometer scale are required to design materials for structural use. Although the scanning electron microscope (SEM) has been used for imaging from low to high magnification, there remained a gap in information between SEM and transmission electron microscope (TEM) observation. In most cases, these two techniques have been used independently. Thanks to the improvement of spatial resolution at low-voltages and the implementation of multiple detectors, SEM today has been enhanced to provide richer information on the microstructure rather than simply giving morphological information. We have been emphasizing the importance of SEM signal "acceptance". By optimizing both the landing energy and working distance (WD), secondary electron (SE) images can give either chemistry or morphology information and backscattered electron (BSE) images can extract either atomic number or channeling contrast. In this paper, we will present interactive applications of SEM and TEM for the characterization of precipitates as well as the second phase in steels.

A precise characterization of precipitates is crucial because these precipitates are utilized to control steel mechanical properties. Regarding different types of carbides appearing in heat resistant Cr-Mo steels, their identification required characteristic X-ray and diffraction analysis because it is usually not possible to differentiate carbides from their size and morphology alone. With an SEM we have found that low accelerating voltages with a short working distance enables differentiation of four types of carbides and the AlN precipitates. A small penetration depth of the primary electron and selective acquisition of type 1 SE is the key to extracting material contrast. This technique will allow quantification of size, distribution and area fraction of each precipitate for a large area using a bulk specimen.

Complex phase steel enables both high strength and high ductility, thus a quantitative estimation of microstructure is vital. We proposed a method for selective visualization of martensite, which is a hard component of complex phase steel, by controlling the collection angle of BSE. After collecting high angle (measured from surface) BSE, thus visualizing a small fraction of martensite in a thin foil specimen, the same area was observed through TEM for further investigation. Combined use of SEM, TEM and transmitted-EBSD enabled quantitative and detailed characterization of martensite that contains a small proportion of austenite. The rich information obtained by SEM with an acceptance control will give a clear guide for further TEM characterization. The combined use of SEM and TEM will lead to new material design.