We studied the behaviors of contrast in backscattered electron (BSE) images of cross-sectional heat-treated steel under various accelerating voltages and take-off angles. Changes in these conditions resulted in dramatic changes in contrast. Low accelerating voltage and low take-off angle improved the surface information and channeling contrast, whereas high accelerating voltage and high take-off angle enhanced the bulk information and reduced channeling contrast, resulting in improved $Z$ contrast. Such behavior can be understood by the ratio of low-loss electrons (LLEs), which are related to channeling contrast, to the inelastic BSE components detected. The distribution of these components varies with the accelerating voltage and take-off angle: the detection ratio of LLE to inelastic BSE increases with decreasing accelerating voltage and take-off angle. The results obtained in this study will be useful for obtaining and crystallographic information separately in BSE images for the material of interest.

KEY WORDS: microstructure analysis; scanning electron microscopy; backscattered electron; accelerating voltage; take-off angle; channeling contrast; $Z$ contrast.

1. Introduction

Backscattered electron (BSE) imaging is widely used for structural observation of various materials, because the images contain various information such as mean atomic number ($Z$), crystal orientation and topography. The origin and mechanism of these contrasts have been already discussed. One of the most important contrast mechanisms of BSE is the $Z$ contrast, in which the backscattering coefficient depends on the mean atomic number $Z$, which allows heavier materials to be recognized as brighter intensity. Meanwhile, the backscattering coefficient also depends on the orientation of the incident electrons and the lattice planes of crystalline specimens, due to the diffraction effect of low-loss electrons (LLEs) in BSEs at the lattice planes. This allows grains with different orientations in polycrystalline specimens to be imaged as channeling contrasts. The channeling effect reflects information only in a thin surface layer, the thickness of which is of the order of the absorption length of the primary Bloch-wave field, i.e. a few nanometers.

Recently, SEM systems with new BSE detectors have opened a new window on BSE observations: BSE images with $Z$ information amplified on the surface can be measured using energy-selected high take-off angle BSEs at ultra-low accelerating voltages of less than 1 kV. Nanometer-sized precipitates have been observed by low take-off angle BSEs even at the accelerating voltage of 15 kV, the penetration depth of which is much deeper than the size of the precipitates. Taniyama explained that the mechanism of the latter phenomena is caused by the suppression of resolution degradation, because BSEs at low take-off angle are emitted from a very thin surface layer.

It is important to understand the origin and mechanism of these contrasts in order to use BSE images for advanced structural analyses. However, there have been few systematic studies on contrast changes by accelerating voltage and take-off angle.

In this study, contrast behavior was studied by independently changing the accelerating voltage and take-off angle. Based on the results, we propose optimum conditions for BSE image acquisition for $Z$ contrast and channeling contrast, respectively.

2. Experimental

The SEM systems used in this study were Ultra 55 or Ultra Plus manufactured by Carl Zeiss NTS GmbH. These systems have two BSE detectors, one at the objective lens and the other in the column, as shown in Fig. 1. The angle-selective BSE (AsB) detector mounted at the objective lens can control the take-off angle of BSEs by changing the working distance (WD). Meanwhile, the energy-selective BSE (EsB) detector mounted in the column detects high-angle BSEs, and detection energies of the BSEs are filtered...
by controlling the grid bias in front of the detector. BSE images were observed with these detectors at the accelerating voltage of 2 kV, 5 kV and 15 kV, and changing the WD from 1.5 to 15 mm. The take-off angles of the AsB detector were estimated to be 27–36°, 39–49°, 50–60°, 69–75° (2 kV) and 73–78° (5 kV and 15 kV) from the geometry of the detector and the specimen. The take-off angle of the EsB is estimated to be approximately 90°, which does not change significantly by WD. During the observations of EsB images, the WD was fixed to 3 mm, and the grid bias was set to 1.5 kV in order to eliminate low-energy secondary electrons. To minimize contamination on the specimen, the observation areas were changed for each accelerating voltage.

The specimen used was heat-treated low carbon steel with an oxide layer mainly composed of magnetite (Fe₃O₄). A cross-sectional specimen was prepared as follows: a piece of the sample was embedded in a conductive resin and then polished with a final polishing colloidal silica suspension (OP-U, Struers).

This specimen had the following features. It consisted of a 10 μm-thick oxide layer mainly composed of magnetite and steel substrate, which had different mean atomic number $Z$. Both layers were polycrystalline materials. Small iron particles were embedded in the scale layer. Changes in $Z$ and channeling contrast are discussed using BSE images from oxide and matrix areas and crystal orientation, respectively. Information depth is also discussed by measuring changes in the contrast of iron particles in the oxide layer.

3. Results

The dependence of contrast in BSE images on take-off angle was studied at the accelerating voltage of 15 kV, which is generally used in many observations. The results are shown in Fig. 2. Regarding the changes in contrast of the steel grains A and B and the magnetite grains C and D, the channeling contrast among the same materials decreased by increasing the take-off angle. Images composed of mainly $Z$ information when the take-off angle became higher than 50–60° are shown in Figs. 2(c)–2(e). Because the brightest crystal grain C in the oxide layer with lower $Z$ was darker than the darkest crystal grain B in the steel substrate with higher $Z$ value in these figures, those images can be explained to be $Z$ contrast rich. Meanwhile, the take-off angle of BSEs also affected the depth from which BSEs were measured. Some Fe particles existing near the surface that were visible at the lower take-off angle disappeared when a higher take-off angle was chosen. Other Fe particles embedded in the scale became visible as the take-off angle became higher, as indicated by the arrows in Fig. 2. In this way, the information depth of BSE images became deeper as the take-off angle increased.

Figure 3 shows the changes of contrast in BSE images by take-off angle at the lower accelerating voltage of 5 kV. At this voltage, the brightest grain D in the oxide layer was

![Fig. 1. Schematic illustration of the position and take-off angle of BSE detectors (EsB and AsB) in the SEM systems used in this study. The take-off angle of the EsB detector was larger than that of the AsB detector.](image)

![Fig. 2. Cross-sectional BSE images obtained from heat-treated steel by the AsB ((a)–(d)) and EsB((e)) detectors at the accelerating voltage of 15 kV. Images (a)–(d) were taken by the AsB detector and (e) by the EsB detector. The take-off angles were (a) 27–36°, (b) 39–49°, (c) 50–60°, (d) 73–78° and (e) approximately 90°.](image)
brighter than the darkest grain A in the steel substrate at the lower take-off angle as shown in Figs. 3(a)–3(c). This difference was more prominent than that at 15 kV, which can be explained as the BSE images at 5 kV being more enriched with channeling contrast than those at 15 kV. When we compare the changes in contrast of the steel grains A and B and the magnetite grains C and D, the channeling contrast among each material decreased with increasing take-off angle. The images were composed of mainly Z information when the take-off angle became higher than 73–78°. These angles are higher than those when the accelerating voltage was 15 kV as shown in Figs. 3(d) and 3(e). Furthermore, the topologies at grain boundaries or the interface between the oxide layer and the substrate were observed at the lowest take-off angle as shown by the arrow in Fig. 3(a). This indicates that a low take-off angle makes BSE images more sensitive to topographic information.

Figure 4 shows the changes of contrast in BSE images with take-off angle at the accelerating voltage of 2 kV. Although the channeling contrast gradually decreased by increasing the take-off angle, the channeling contrast was still strong compared with 15 kV and 5 kV and the channeling and Z contrast superimposed on the BSE image even at the highest take-off angle of 90°.

From these results, the relationship between the accelerating voltage, take-off angle and contrast in the BSE image can be explained as shown in Fig. 5. The Z contrast is more enriched than channeling contrast at 15 kV. By lowering the
The characteristics of the BSE contrast at each accelerating voltage can be summarized as follows.

1) The observation of BSE images at 15 kV is appropriate for bulk materials with information rich. Crystallographic information can be superimposed on the images at lower take-off angle.

2) At 5 kV, we can select crystal orientation information rich or information rich by controlling the take-off angle.

3) The BSE images become crystallographic rich and surface sensitive at 2 kV. Information can be superimposed on the images at higher take-off angle.

Thus, it is very important to choose the accelerating voltage and take-off angle depending on the purpose of the surface structural observations such as information depth and crystal orientation and information.

### 4. Discussion

The relationship between accelerating voltage, take-off angle and contrast of BSE images is discussed to clarify the origin of the contrast change as described in section 3.

BSEs consist of LLEs and inelastic BSEs. LLEs include electrons without energy loss and those that slightly lose energy by primary electrons. Inelastic BSEs lose energy by diffusion in a specimen. Channeling contrast is caused by the differences between the backscattering coefficients of each material, and is induced by both LLEs and inelastic BSEs. Channeling contrast is caused by orientation anisotropy of the backscattering coefficient, which is due to the diffraction effect of LLEs on crystalline materials. Berger et al. demonstrated that anisotropies of LLEs cause channeling contrast by measuring the angular distribution of energy spectra of BSEs on various single crystalline materials using a 4-axis movable compact concentric hemispherical analyzer mounted in the chamber. Consequently, the changes of contrast in BSE images such as and channeling contrast can be roughly explained by the ratio of LLEs to inelastic backscattered electrons.

Figure 6 shows a schematic illustration of the detectable areas of LLEs and inelastic BSEs. The diffusion area of inelastic BSEs is based on Archard’s diffusion model, in which electrons diffuse in a sphere centered at x below the surface with radius R-x as shown in Fig. 6, where R is electron range and x = 40R/7. The diffusion area of inelastic BSEs detected is the circular cone shown in Fig. 6. The diffusion area of LLEs is assumed to be a hemisphere centered on the point where the incident beam approaches the surface with a radius of one half of the inelastic mean free path (IMFP) of electrons. The relationship between the volume ratio of LLE to inelastic BSE and accelerating voltage is plotted in Fig. 7. IMFP and electron range are calculated from the predictive formulas TPP-2M and the empirical formula of Kotera et al., respectively. Although it is necessary to consider crystal orientation, the specimen is assumed to be non-crystalline iron. The volume ratio of LLE gradually increases as the accelerating energy is decreased, and shows a dramatic rise below 5 kV. However, the diffusion area of BSE does not match the intensity of BSE, because BSE loses energy and decreases intensity by the diffusion effect in the specimen.

Angular distributions of BSE energy spectra have been reported in several articles. The relationship between the intensity of a sharp LLE peak and the following smooth inelastic BSE peak changes with take-off angle. The ratio of integrated intensity of LLE to inelastic peaks decreases with increasing take-off angle. This means that the escape depth
of BSE which is detected is changed by the take-off angle. The intensity ratio of LLE becomes higher when the take-off angle is lower, because the escape depth of the detected BSE becomes shallower. Meanwhile, the intensity ratio of inelastic BSE becomes higher when the take-off angle is higher, because the escape depth of the detected BSE becomes deeper.

Thus, it is roughly understood that changes of contrast in BSE images are caused by changes in the ratio of LLE in the detected BSE signal. For a more accurate discussion, it is necessary to consider the following matters: (1) The models of electron scattering and diffusion taking into account the effects of crystal orientation and mean atomic number, and (2) Estimation of the intensity taking into consideration the sensitivity of BSE at different energies.

Using the relationship between accelerating voltage, take-off angle and contrast in BSE images such as \( Z \) and channeling contrast, we can easily observe BSE images under optimum conditions for observation. The way to choose the optimum conditions for observation is as follows. When we observe a specimen under general conditions such as accelerating voltage of 15 kV and WD of 10–15 mm, information on the crystal orientation and mean atomic number is blended on the BSE image. Instead, we observe at the accelerating voltage of 5 kV, which is the optimum voltage for selecting the crystal orientation and mean atomic number information. Then, we observe at lower \((39–49^\circ)\) and higher \((\text{approximately} \, 90^\circ)\) take-off angles to enhance the crystal orientation and mean atomic number information, respectively. As shown in Fig. 8, we can measure two BSE images enriched each channeling and \( Z \) contrast at the same time with two different BSE detectors having different take-off angles.

5. Conclusion

We studied the behaviors of contrast in BSE images of cross-sectional heat-treated steel under various accelerating voltages and take-off angles. High accelerating voltage enhances bulk information and \( Z \) contrast, whereas low accelerating voltage improves surface information and channeling contrast. High take-off angle also enhances bulk information and \( Z \) contrast, whereas low take-off angle improves surface information and channeling contrast. Changes in these conditions resulted in dramatic changes of contrast on BSE images.

We propose that BSE images should be measured under optimum conditions for the material of interest as follows: (1) Observation at 15 kV is appropriate for bulk materials with \( Z \) information rich; crystallographic information can be superimposed on the images at lower take-off angle. (2) At 5 kV, we can select crystal orientation information rich or \( Z \) information rich by controlling take-off angle. (3) Observation at 2 kV is appropriate for surface sensitive and crystallographic information rich; \( Z \) information can be superimposed on the images at higher take-off angle.

These behaviors can be understood by the ratio of LLE to inelastic BSE components, which are detected. The distribution of these components varies depending on the accelerating voltage and take-off angle: the ratio of LLE to inelastic BSE increases with decreasing accelerating voltage and take-off angle, thus enriching the channeling contrast.

The results obtained in this study will be useful for obtaining mean atomic number and crystallographic information separately in BSE images for the material of interest. The technique proposed in this study can be combined with the technique to separate topographic and material information on secondary electron images\(^{12–14} \) to assist the design and development of steel materials based on microstructural characterization.

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