Phase Analysis on Dual-Phase Steel Using Band Slope of Electron Backscatter Diffraction Pattern

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Abstract: A quantitative and automated phase analysis of dual-phase (DP) steel using electron backscatter diffraction (EBSD) was attempted. A ferrite–martensite DP microstructure was produced by intercritical annealing and quenching. An EBSD map of the microstructure was obtained and post-processed for phase discrimination. Band slope (BS), which was a measure of pattern quality, exhibited much stronger phase contrast than another conventional one, band contrast. Owing to high sensitivity to lattice defect and little orientation dependence, BS provided handiness in finding a threshold for phase discrimination. Its grain average gave a superior result on the discrimination and volume fraction measurement of the constituent phases in the DP steel.

Key words: phase analysis, electron backscatter diffraction, pattern quality, band slope, dual-phase steel, martensite

INTRODUCTION

Recently, various high-performance steels are being developed and most of them are based on multiphase microstructures. The products classified as advanced high-strength steels (AHSS) and their derivatives are the representative examples for this trend (Han et al., 2009; WorldAutoSteel, 2009). In tailoring the properties of these products, control on the volume fractions of constituent phases is the primary consideration. Therefore, a convenient and precise tool for phase analysis is an essential prerequisite.

Dual-phase (DP) steel is currently the most commercialized grade among AHSS. It shows excellent strength–toughness balance, bake hardenability, planar isotropy, low-yield ratio compared with the conventional high-strength low-alloy steels and also exhibits nonaging characteristics at room temperature (Matsuoka & Yamamori, 1975; Nakaoka, 1982, 1984; Llewellyn & Hills, 1996; Han et al., 2008, 2010). Its microstructure consists of ferrite matrix and martensite particles, the latter of which gives the product a high strength. The body-centered tetragonal lattice of martensite generally shows little difference from the body-centered cubic lattice of ferrite, consequently causing a large difficulty and complication in their discrimination when using conventional diffraction-based methods.

Electron backscatter diffraction (EBSD), which enables the direct correlation of the crystallography to the microstructure, is a versatile tool in the characterization of microstructures (Randle & Engler, 2000; Schwartz et al., 2000; Maitland & Sitzman, 2007) and has currently become a common tool. With regard to the phase analysis of DP steels, a few studies have been performed by utilizing

the quality of pattern that was evaluated by the signal intensities of the bands in the EBSD pattern (Wilson et al., 2001; Wu et al., 2005; Ryde, 2006; Wright & Nowell, 2006; Nowell et al., 2008; Kang et al., 2011). As martensite contains much more lattice defects introduced during transformation, it emits poorer quality patterns than ferrite. Thus, the two phases can be discriminated using the quality parameters of pattern. In this study, band slope (BS), which can represent the quality of EBSD pattern (EBSP), is examined for detection and volume fraction measurement of martensite in DP steels. It is also compared with band contrast (BC), which is the conventional quality parameter of EBSP.

MATERIALS AND METHODS

The chemical composition of the DP steel is presented in Table 1. The cold rolled sheet was annealed at 800°C in a salt bath for 5 min and water-quenched, which produced the ferrite–martensite DP microstructure. The specimen for EBSD mapping was prepared by electropolishing. In this study, an HKL system by Oxford Instruments was used for recording and indexing of EBSD patterns. The acceleration bias and the probe current of electron beam were 20 kV and 4 nA, respectively. The phase analysis procedure, which is described in the following, was performed on a map of 500 × 600 pixels in 0.1 μm step size.

The post-processes of the raw data such as noise filtering, grain identification, and grain averaging were carried out following the procedure developed by the author (Kang et al., 2011). As the criterion for phase discrimination, BC and BS were separately examined. It should be noted that BC is equivalent to the quality parameters in other commercial EBSD systems, that is, image quality or pattern quality

Table 1. Chemical Composition of the Dual-Phase (DP) Steel Used (wt%).

C	Si	Mn	Cr	P	N	S
0.08	0.10	1.8	0.20	0.02	0.004	0.003

(Wilson et al., 2001; Wu et al., 2005; Ryde, 2006; Wright & Nowell, 2006; Nowell et al., 2008). On the other hand, BS is a less common quality parameter that is evaluated by the maximum gradient of intensity at the edge of the bands in the pattern (Maitland & Sitzman, 2007). The difference between the two parameters is schematically shown in Figure 1. Across the width of a band in EBSD of Figure 1a, BC can be regarded as the relative signal intensity compared with background level, and BS as the slope of intensity at the edge as shown in Figure 1b. Therefore, a high-quality pattern shows bright bands (high BC) with sharp edges (high BS). It should be noted that the actual formulation of the two types of parameters can be varied depending on EBSD systems and their evaluation is performed on the Hough transformed pattern.

Figure 1. Evaluation of band contrast (BC) and band slope (BS): (a) a band in an electron backscatter diffraction pattern indicated by white broken lines, (b) signal intensity profile across the band width at an arbitrary location and the definition of BC and BS.

RESULTS

In Figure 2, the raw mapping data are presented. The BC map of Figure 2a clearly exhibits the characteristics of DP structure, which enables the manual discrimination of phases. However, the difference in BC between ferrite and martensite is not marked enough. Thus, a distinct threshold of BC cannot be yielded in the profile of Figure 2b. On the other hand, the BS map shows an even stronger image contrast as

Figure 2. Dual-phase microstructure revealed by electron backscatter diffraction: (a) band contrast (BC) map, (b) profile of BC, (c) band slope (BS) map, and (d) profile of BS (inset is a magnified subset).

Figure 3. Effect of grain averaging: (a) grain-averaged band contrast (BC) map, (b) profile of grain-averaged BC, (c) grain-averaged band slope (BS) map, and (d) profile of grain-averaged BS (inset is a magnified subset).

shown in Figure 2c. Only boundaries and parts of martensitic regions are revealed by darker tone, as they have low BS values. In the BS profile of Figure 2d, owing to the strong contrast, only a sharp single peak of unit width at the maximum value of 255 is visible in the full measured range. By setting the BS value of 255 as the threshold, a quick and crude estimation of phase volume fraction is possible. However, inside the martensitic region, some pixels have the maximum BS value, whereas the ferritic region near the boundaries exhibits lower values. This should result in considerable inaccuracy in discrimination, that is, irrational separation of a single structural unit (lath, block, or grain) into different phases. To avoid this problem, the grain average of BC and BS is utilized in Figure 3. During grain averaging, the pixels adjacent to boundaries are disregarded to minimize the influence of boundary or grain size.

By grain averaging, all constituent pixels of a grain have the same value, which leads to the loss of detailed intragranular morphology as shown in Figures 3a and 3c. However, it aids in finding a threshold value for phase discrimination in the BC profile of Figure 3b. The value of 56 at which a valley forms in the profile can be used. In the profile of grain average BS, the extraordinarily sharp peak in Fig-

Figure 4. Reconstructed phase map (■: ferrite, ■: martensite) by the grain average of (a) band contrast and (b) band slope.

ure 3d is retained but became a little bit smoother as shown in Figure 3d. A threshold of 248 can be recognized, although there is a very small ambiguity.

With the two kinds of threshold, phase discrimination was performed, respectively, and the result is shown in Figure 4. It can be seen that the BC threshold is not adequate as shown in Figure 4a, and BS one presents a reasonable result as in Figure 4b. The measured volume fractions of martensite are 7.7 and 32.9%, respectively.

DISCUSSION

The amount of lattice defects in martensite depends on its carbon (C) content. When martensitic volume increases in a steel of fixed composition, the enrichment of C in martensite decreases, resulting in reduced lattice strain during transformation. The quality of EBSP from the martensite increases. Therefore, the observed failure of phase discrimination in Figure 4a is primarily caused by the small difference in the perfectness of lattice between ferrite and martensite. Only a limited number of fine martensite grains in which the local enrichment of C is severe can be recognized. A higher value of grain average BC is recommended as the threshold, but it is difficult to unambiguously find one in the profile of Figure 3b. In the present case, the sensitivity of BC to the lattice perfectness is not sufficient, although it is also considerably shaded by the orientation contrast of BC. If the specimen was annealed at a lower temperature, consequently containing a smaller volume of martensite with larger defect density, BC would be effective in phase discrimination.

Contrary to BC, BS is sensitive enough to capture the small difference in the lattice completeness, in addition to showing negligible orientation dependence. Therefore, it provides much stronger phase contrast for DP steels, which helps to find a pronounced threshold for phase discrimination. Using its grain average value gives very sound results of discrimination and volume fraction measurement as shown in Figure 4b. When the annealing temperature, consequently the volume fraction of martensite varied for the provided steel specimen, the above threshold value of grain average BS did not lose the consistency nor the validity in the result.

CONCLUSIONS

Two parameters of EBSP quality were tested for phase analysis of a DP steel. It was shown that BS exhibited better phase contrast than the conventional parameter, BC. After grain averaging of the quality parameters, the threshold for the phase discrimination could be obtained and the automatic judgment of the phases of structural units was possible. As anticipated from its superior phase contrast, BS yielded a much better result in the quantitative phase analysis. The higher sensitivity to lattice defect and little orientation contrast provided BS with the better suitability to the discrimination of martensite from ferrite. The effectiveness would be remarkable when the crystal completeness of martensite approaches that of ferrite.

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