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Estimation of phase fraction in dual phase steel using microscopic characterizations and dilatometric analysis

Jun-Yun Kang[a], Seong-Jun Park[a], Dong-Woo Suh[b,*], Heung Nam Han[c]

[a]Korea Institute of Materials Science, 797 Changwon-daero, Changwon, Gyeongnam 642-831, Republic of Korea
[b]Graduate Institute of Ferrous Technology, Pohang University of Science and Technology, 77 Cheongam-ro, Nam-gu, Pohang, Gyeongbuk 790-784, Republic of Korea
[c]Department of Materials Science and Engineering, Seoul National University, 599 Gwanak-ro, Gwanak-gu, Seoul 151-744, Republic of Korea

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ABSTRACT

We made a quantitative comparison of three different methods, optical microscopy by tint etchant, EBSD phase mapping and dilatometry for analysis of phase fraction in steel subjected to intercritical annealing and isothermal treatment. While the results from optical microscopy and EBSD technique showed quantitative agreement, the dilatometry gave rather higher martensite fraction compared to the microscopic analyses. Nevertheless, all three methods showed qualitative agreement in the variation of martensitic fraction depending on the processing conditions. The analyses revealed that the martensitic fraction in final microstructure decreased as intercritical annealing temperature increased because it deteriorated the hardenability of austenite. Raising the isothermal treatment temperature increases the martensite fraction due to the increases of austenite fraction, which transformed into martensite afterward.

1. Introduction

Recently, multi-phase microstructures are favored to meet the high performance requirements in steel products. The automotive steel sheets classified as advanced high strength steels (AHSS) [1] will be a representative example. In tailoring the mechanical properties, controlling the fractions of constituent phases is one of the critical issues, which requires reliable tools for phase analysis.

In the analysis of steel microstructures, the phases which need to be discriminated and quantified will be austenite and its transformation products. The latter includes the three major phases of ferrite, bainite and martensite according to transformation temperature and mechanism. Each of them also has a few sub-classes of characteristic morphology and crystallography. These diverse microstructures from austenite decomposition make the phase analysis of steels the most complicated among modern engineering alloys. As austenite has the distinct lattice structure of face-centered cube (FCC) compared to its decomposed products whose lattices are primarily based on body-centered cube (BCC), it can be distinguished with relative ease. Most difficulties arise when discriminating among the microstructures from austenite decomposition because their crystal structures are almost identical. Although martensite has body-centered tetragonal lattice, the deviation from cubicity is at most 9% in extraor-

* *Corresponding author*. Tel.: +82 54 279 9030; fax: +82 54 279 9299.
E-mail address: dongwoo1@postech.ac.kr (D.-W. Suh).

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dinarily high C concentration, e.g. 2 wt% [2]. In most practical situations, it is much smaller. Therefore, attempts on unambiguous discrimination among these phases using the ordinary X-ray or neutron diffraction were not satisfactory [3].

Practical procedure for phase analysis of steels has been a point counting of characteristic morphologies on micrographs. In optical microscopy (OM), various tint etchants help this procedure as they decorate different phases with different colors. Image analysis followed by automated counting is frequently used instead of manual counting although standardization for the image processing is still not established. Recently, there have been continuing trials to use electron backscatter diffraction (EBSD) technique for phase analysis in steels [4–9]. As EBSD stands on diffraction technique as well, it has the aforementioned difficulty in handling the phases decomposed from austenite. Symmetry in the EBSD pattern cannot resolve the little differences in the lattice structures with satisfactory precision. However, it also provides morphological information as well as crystallographic one as various forms. Earlier works pointed out the usefulness of the pattern clarity [4–7]. The quantitative measure of this is provided with several terms such as pattern quality [4,10], image quality [5,6,8] and band contrast [11] depending on the EBSD system while they all have equivalent physical meaning. As the phases decomposed from austenite have different levels of defect density introduced during transformation, they will show different levels of pattern clarity. This was effectively used for phase identification in dual phase (DP) steels [4,5,8,9] and expected to be useful in some multi-phase steels [5,9] although it still needs further improvement. Meanwhile, dilatometry has served to estimate the fraction of constituent phases [12–15]. It monitors the length change of specimen during a given thermal cycle. Due to the atomic volume change during transformation which is reflected on the length change, the change of phase fraction during a thermal cycle can be analyzed quantitatively. Although this method does not provide information on microstructure, it gives the phase fraction as a function of temperature and time, by which the transformation kinetics can be evaluated as well.

In this study, it was aimed to make a comparative study on the aforementioned three methods for phase analysis. For this purpose, a dual phase (DP) steel sheet was prepared as the test material. The microstructure of DP steel is mainly comprised of ferrite and martensite, and it is the most commercialized product among AHSSs, which makes the discussion on phase analysis important in practical sense.

2. Experimental

Chemical composition of a cold rolled DP sheet steel was 0.06C–0.1Si–2.0Mn. To obtain the dual phase structure, inter-critical annealing followed by controlled cooling and isothermal treatment was performed, which simulates a commercial continuous annealing line including the intermediate heating for galvanic coating. The heat treatment was performed with dilatometer (Dilatronic III, Theta Inc.) using 3 mm-wide and 10 mm-long specimens with a longitudinal direction parallel to the rolling direction. The thermal cycle is

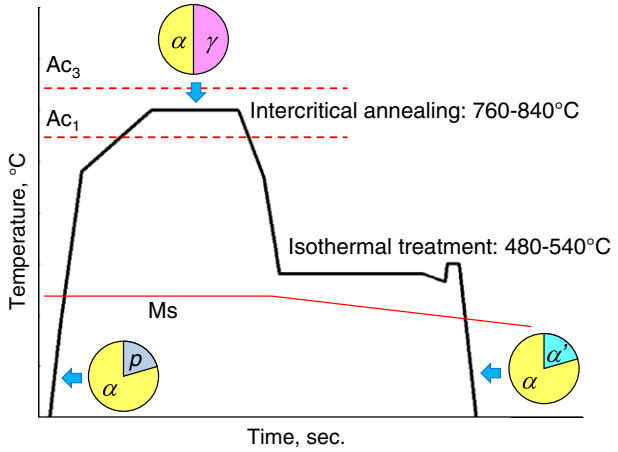

Fig. 1 – Thermal cycle of the continuous inter-critical annealing and the constitution of microstructure (α: ferrite, γ: austenite, α′: martensite, p: pearlite).

illustrated in Fig. 1 with schematics of microstructural constitution. Note that the detailed heating and cooling rates are proprietary information. The initial microstructure is a mixture of ferrite (α) and pearlite (p), which is converted into ferrite and austenite (γ) during intercritical annealing. In the course of the first cooling section prior to isothermal treatment, part of γ reverts to α. During the final cooling section after the intermediate heating, the remaining γ transforms into martensite (α′); consequently the final microstructure of ferrite and martensite (α + α′) is attained. To investigate the influence of process condition on the phase fraction, the intercritical annealing temperature was varied as 760, 800 and 840 °C with fixed isothermal treatment temperature of 480 °C, or isothermal treatment temperature was changed as 420, 480 and 540 °C for given intercritical annealing temperature of 800 °C.

Standard metallographic procedure was employed to evaluate the martensite fraction using optical microscopy (OM) and EBSD. In analysis with OM, polished specimens were etched with 2% nital solution, then dipped into a tint etching solution [16–18] for 5–10 seconds. The latter was a mixture of 3% aqueous sodium metabisulfite and 4% picric acid in ethyl alcohol in a 1:1 ratio. Optical micrographs were taken under white light. For phase mapping using EBSD, the specimen surface was electrolytically polished by Struers Lectropol-5. The electrolyte was the solution of perchloric acid and ethanol with the volume ratio of 1:9, cooled to −20 °C. The operating bias, the duration and the flow rate were 31 V, 25 seconds and 20, respectively. EBSD mapping was performed using field emission type SEM (JSM-7001F, JEOL) equipped with Channel 5 EBSD system and Nordlys-F camera (Oxford Instruments). The measure of pattern clarity was represented by band contrast (BC) in this system. The size of mapping area was 400 × 500 with 0.2 μm step of square grid. The acceleration voltage and the probe current of electron beam were 20 kV and 4 nA, respectively. And the camera was operated in 168 × 128 pixel dimension (8 × 8 binning). The input lattices for EBSP indexing were FCC and BCC.

For the analysis of phase fraction using dilatometric data, detailed procedure is described in the next section.