

High precision FE-EPMA for carbon distribution mapping during ferrite transformation in in low carbon Mn-Si steels

Kaneharu.Okuda¹, Takako Yamashita¹, Tatsuya Nakagaito²

¹Steel Research Laboratory, JFE Steel Corporation, 1 Kawasaki-cho, Chuo-ku, Chiba, 260-0835 Japan

²Steel Research Laboratory, JFE Steel Corporation, 1 Kokan-cho, Fukuyama, Hiroshima, 721-8510 Japan

Complex microstructures in advanced high-strength steel sheets such as TRIP steels are controlled by the heat treatment conditions in the production process. Although the carbon content in retained austenite is a key factor for the stability of retained austenite in TRIP steels, a direct method for evaluating the distribution of the carbon content in microstructures with high accuracy has been required. JFE Steel has developed a unique FE-EPMA which is capable of evaluating the carbon distribution in microstructures. In this study, this method was applied to study the change in carbon enrichment during the annealing process of formable high-strength steel sheets. The ferrite transformation behavior during the cooling process changed greatly depending on the holding temperature in the two-phase region. When heat treatment was carried out on the higher temperature side of the intercritical region, the ferrite transformation proceeded remarkably compared with the low-temperature holding condition. Mn, which is a substitutional element, was unable to move in the NPLE mode, and only carbon was partitioned. In this way, it is shown that carbon distribution behaviors of this type can be analyzed in detail by this carbon analyzer.

Keywords: carbon analyzer, ferrite transformation, EPMA, annealing

1. Introduction

In the production of high-performance high-strength steel sheets, in addition to optimization of the chemical composition, the development of improved heat treatment technology has also made it possible to control complex microstructures in steel.^{1,2)} In high-ductility TRIP steels, it is necessary to stabilize retained austenite in the annealing process. Although the carbon content in austenite is an important factor for the stability of retained austenite, a direct method for evaluating the distribution of the carbon content in the microstructures with high accuracy has been required. Although element maps were usually obtained by field emission electron probe microanalysis (FE-EPMA), quantification of the carbon content was not possible due to hydrocarbon contamination. With conventional techniques, the lower limit of quantification of carbon had been limited to around 0.1 mass%, even in point analysis (Fig. 1). Recently, JFE Steel developed a unique FE-EPMA method which is capable of evaluating the carbon distribution in microstructures.^{3,4)} Here, this method was applied to track the change in carbon enrichment during the annealing process of formable high-strength steel sheets.

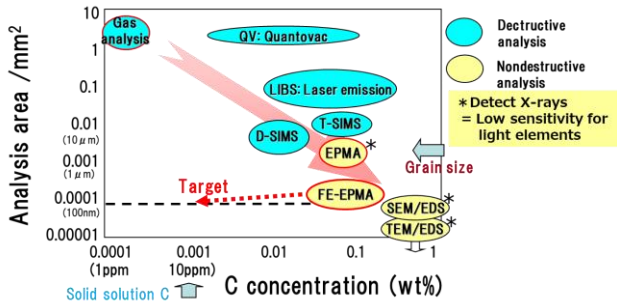


Fig.1 Comparison of carbon analysis methods.

2. Carbon analyzer

In the carbon analyzer, the effect of hydrocarbon contamination was reduced as much as possible to improve the accuracy of carbon analysis even at low concentrations.

Figure 2 shows the effect of the anti-contamination devices. When both plasma cleaning and heating of liq. N₂ gas were applied, the carbon intensity was unchanged even after a number of iterated irradiations. This system is capable of maintaining a lower limit of quantification of carbon of 0.01 % or lower in 2-dimensional analysis. Using this technique, the 2-dimensional distribution of carbon corresponding to the microstructures in steel sheets was successfully visualized for the first time.

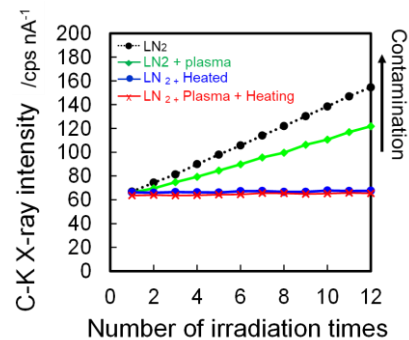


Fig.2 Effect of anti-contamination devices; increment of X-ray counts with electron beam irradiation.

3. Experimental procedure

The chemical composition of the steel used in the present study was 0.17%C-1.5%Si-1.7%Mn. Ae1 and Ae3 were 708 °C and 857 °C, respectively. Vacuum-melted steel was cast into a 50 kg ingot, followed by hot-rolling to a 27 mm thick slab. The slab was reheated at 1 250 °C and then hot-rolled to a sheet thickness of 4.0 mm with a finishing temperature of 900 °C, followed by holding at 600 °C for 1 h. The surface of the hot-rolled sheet was ground to a thickness of 3.2 mm. After cold rolling to a thickness of 1.2 mm, heat treatment was performed with a salt bath furnace. The specimen was water-quenched from each temperature during gas cooling at a cooling rate of 10 °C/s. The holding temperature (Ta) was set to 740 °C or 800 °C.

Microstructural observation and FE-EPMA analysis of

the specimens were carried out as follows. The microstructure was characterized by scanning electron microscopy (SEM) using specimens etched with nital. The distributions of C, Mn and Si in the microstructure were measured using the new FE-EPMA technique.^{3,4)} First, the distribution of C was measured while preventing contamination of the surface. The accelerating voltage V_e and the probe current I_e of the primary electron beam of the C measurement were 7 kV and 5 nA, respectively. In order to obtain a larger S/N ratio in the measurements of Mn and Si, V_e and I_e were changed to 9 kV and 10 nA, respectively. Pure Fe and reference steels with C contents between 0.089 and 0.680 mass% were used as standard specimens to quantify the C content. The Mn and Si contents were quantified by the ZAF correction method⁵⁾ using pure Mn and Si as standard specimens.

4. Result and discussion⁶⁾

Figure 3 shows the dependence of the volume fraction of the ferrite phase on the quenching temperature T_q . For $T_a = 800\text{ }^\circ\text{C}$ (solid circles), the fraction is 0.35 at $T_q = 800\text{ }^\circ\text{C}$, and monotonically increases with decreasing quenching temperature. The volume fraction of ferrite is particularly sensitive to T_q between 700 and 600 $^\circ\text{C}$, and finally reaches 0.67 at 400 $^\circ\text{C}$. On the other hand, at $T_a = 740\text{ }^\circ\text{C}$ (open squares), the fraction is close to 0.67, being independent of T_q . Thus, there is a difference in the dependence of the volume fraction of fraction ferrite on T_q according to the holding temperature in the two-phase region. When heat treatment was carried out on the higher temperature side of the two-phase region, the ferrite transformation proceeded remarkably compared with the low-temperature holding condition.

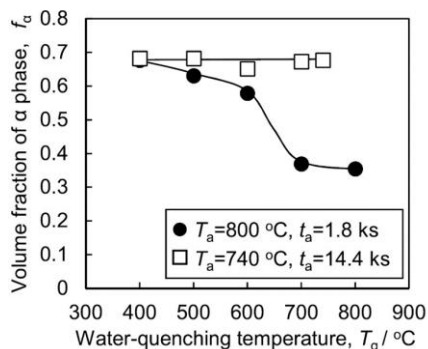


Fig. 3 Change in volume fraction of α phase with quenching temperature.

Figure 4 (a-f) shows SEM images and elemental mappings of the solute elements for the specimens with holding at $T_a = 800\text{ }^\circ\text{C}$ or $740\text{ }^\circ\text{C}$ for 1.8 ks. Figs. 4 (a-c) and (d-f) indicate the results for $T_q = 800\text{ }^\circ\text{C}$ and $400\text{ }^\circ\text{C}$, respectively. In Fig. 4 (d-f), at $T_q = 400\text{ }^\circ\text{C}$, the region with a large Mn content does not necessarily match the one with a large C content, as the former is relatively broader than the latter. The corresponding results for $T_a = 740\text{ }^\circ\text{C}$ and holding time $t_a = 14.4\text{ ks}$ are shown in Fig. 4 (g-i). Unlike for $T_a = 800\text{ }^\circ\text{C}$, however, distinct dependencies of the volume fraction of each phase and the content of each solute element on the quenching temperature are not

recognized. This means that the ferrite transformation is suppressed under the conditions at $T_a = 740\text{ }^\circ\text{C}$. In this case, Mn, which is a substitutional element, was unable to move in the so-called NPLE (negligible partitioning local equilibrium) mode⁷⁾, and only carbon was partitioned.

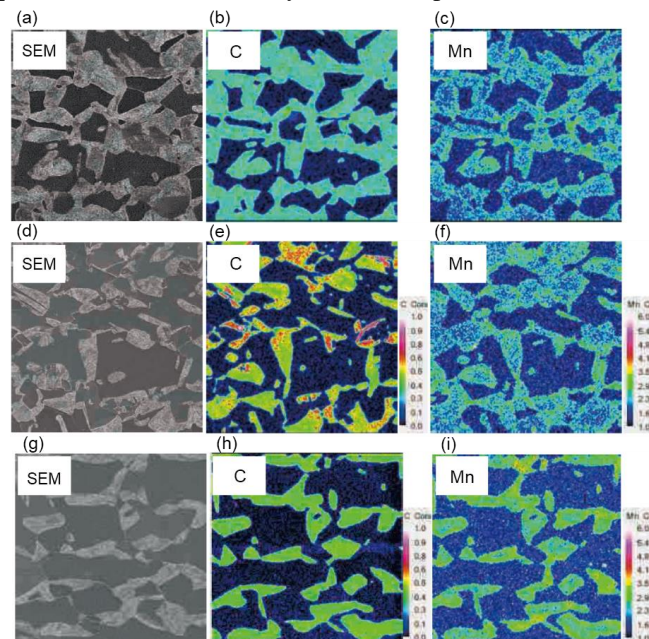


Fig. 4 SEM images and elemental mappings of solute elements for specimens with holding at $T_a = 800\text{ }^\circ\text{C}$ and $740\text{ }^\circ\text{C}$ for 1.8 ks. Figures 4 (a-c) and 4 (d-i) indicate the results for $T_q = 800\text{ }^\circ\text{C}$ and $400\text{ }^\circ\text{C}$, respectively.

5. Summary

The newly-developed high-precision carbon analyzer was applied to track the change in carbon enrichment during the annealing process of formable high-strength steel sheets. This study demonstrated that the carbon analyzer is a useful tool for optimizing the annealing cycle of high-performance high-strength steel sheets.

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