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Analysis Technology of Microstructure Formation in High Performance Dual Phase Steel†

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Abstract:

In order to obtain high performance high strength steel sheets, JFE Steel has developed techniques for analysis of the phase transformation behavior and distribution of elements during intercritical annealing by using diffusion simulation. The phase transformation behavior of steel can be predicted more theoretically by adding computational science to microstructural analysis.

1. Introduction

In recent years, various types of high strength steel sheets have been used in auto bodies to reduce CO_2 emissions by reducing the weight of automobiles. Among these materials, dual phase steel sheets (DP steel) are produced by dispersing martensite in the soft ferrite phase by adding Si and Mn. DP steel possesses outstanding mechanical properties, having a low yield ratio because it does not display yielding characteristics, as well as a large work-hardening coefficient (*n* value) and large elongation. For this reason, DP steel sheets have been applied to auto bodies and are widely used as steel sheets which improve press formability and reduce springback[1,2].

On the other hand, because DP steel has a dual phase structure, it is extremely important to understand the phase transformation behavior that occurs in the microstructure formation process and the distribution of alloying elements to the ferrite phase and martensite phase, and to clarify the relationship with material properties. The metallurgical theory which is the basis of microstructure formation in DP steels is the $\gamma \rightarrow \alpha$ or $\alpha \rightarrow \gamma$ phase transformation. However, because the composition of DP steel is Fe-C-X (where X is a substitution-type alloying element X=X_1, X_2,...), the diffusion rates of carbon and other alloying elements are extremely different, and there are thought to be many cases in which the phase transformation proceeds under local equilibrium or para-equilibrium conditions.

Therefore, JFE Steel developed a technique for analyzing the phase transformation behavior and distribution of elements in intercritical annealing of DP steel, targeting analysis of the phase transformation behavior of DP steel in the manufacturing process of practically used steels. Distribution behavior of the elements in specimens whose microstructure is frozen after $\alpha+\gamma$ intercritical annealing which is the basis of the manufacturing process for cold-rolled DP steel is observed by FE-EPMA (electron probe micro analyzer equipped with a field emission (FE) gun), and is then analyzed by diffusion simulation in the developed analytical technique.

2. Analysis of Microstructural Changes in Intercritical Annealing

2.1 Sample Material and Experimental Method

The sample material used in this study was an Fe-0.125C-1.4 Si-1.98Mn (mass%) for quaternary model allay. In order to evaluate the distribution of Si and Mn in the microstructure formation process, the specimens were frozen in the respective processes of $\alpha+\gamma$ intercritical isothermal transformation and continuous cooling in the manufacturing process for cold-rolled DP steel shown in **Fig. 1**. The variation of the volume fraction of

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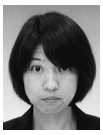

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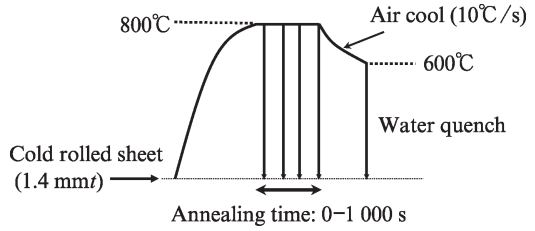

Fig. 1 Schematic diagram of heat treatment [8)]

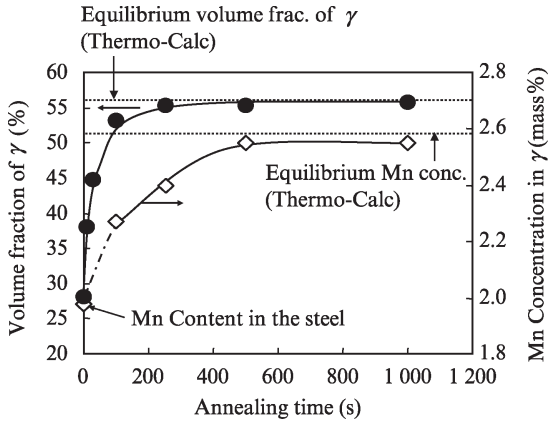

Fig. 2 Variation of the austenite volume fraction and Mn concentration in γ with annealing time at 800˚C [8)]

γ phase and the Mn concentration in the γ phase with holding time during intercritical annealing at 800˚C in the heat treatment process is shown in **Fig. 2**. Although this figure shows the equilibrium of γ phase volume fraction and Mn concentration in the γ phase obtained by Thermo-Calc[3)], the volume fraction of γ phase achieved the equilibrium state more quickly, and distribution of Mn was slower in reaching the equilibrium state.

Therefore, two types of quenched specimens were prepared. Specimen A was held at 800˚C for 100 s so that the volume fraction of γ phase substantially achieved the equilibrium ratio, but distribution of Mn to the γ phase (martensite phase) did not achieve the equilibrium composition. Specimen B was held at 800˚C for 1 000 s so that both the volume fraction of γ phase and the amount of Mn reached the equilibrium composition. After first cooling these two types of steel to 600˚C without quenching, the two specimens were immediately quenched in water. (The quenched specimen of steel A is called specimen C, and the quenched specimen of steel B is called specimen D.)

The elemental distributions of these specimens were measured by FE-EPMA (model JXA-8500 F, manufactured by JEOL Ltd.), and C, Si and Mn were analyzed at the accelerating voltage of 9 kV from the viewpoints of the *S*/*N* ratio and analysis area. The measurements were performed by mapping of a 8000x field of view and quantitative line analysis. Calibration curves obtained with standard substances for trace measurement were used in the quantitative analysis.

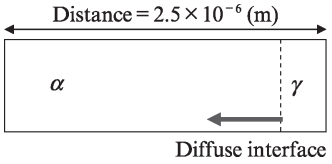

Fig. 3 Geometry models for multi-component diffusion simulation for α→γ transformation at isothermal treatment [8)]

2.2 Analysis of Element Distribution Behavior by Diffusion Simulation

In the diffusion simulation, models for handling various diffusion phenomena were prepared, and the simulation was performed using DICTRA (DIffusion Controlled TRAns-formation), which was developed by the Royal Institute of Technology of Sweden and enables more general analysis[4,5)]. The calculations of phase transformation behavior by DICTRA will be left to the literature[6)]. In JFE Steel, a study by comparison with the experimental values of isothermal transformation behavior was carried out with 0.1C and 0.3C-2.0Mn-Fe (mass%) alloy, and good agreement between the results calculated by DICTRA and the experimental results was confirmed with the 0.1C-2.0Mn-Fe (mass%) alloy[7)].

The calculation model of the isothermal transformation at 800˚C is shown in **Fig. 3**. The initial microstructure of the quaternary model alloy used in the study was α+pearlite. However, as the distributions of Si and Mn in the initial microstructure were almost homogeneous in the EPMA measurements, the transformation from the α phase to the γ phase was calculated under the assumption that the pearlite transforms directly to the γ phase. Based on the fact that the crystal grain size was comparatively coarse, the cell shape was assumed to be rectangular, and the γ phase was modelled as growing from the right side of the cell with the passage of time. As the calculations were limited to the diffusion phenomenon without treating nucleation, an extremely thin (1×10^{-9} m width) γ phase was set initially at the right side of the cell. Assuming the total cell length of 2.5 μm, which is 1/2 of the mean grain diameter of the γ phase, the isothermal transformation at 800˚C was calculated until the time of 1 000 s.

3. Microstructural Analysis of Dual phase Steel[8)]

In order to evaluate the element profiles at the α/γ phase interface in specimens A and B in detail, the amounts of C, Si and Mn were determined by line analysis. The composition (BSE) images at the line analysis position and the results of the quantitative line analysis are shown in **Fig. 4**. First, in the profiles of specimen A,

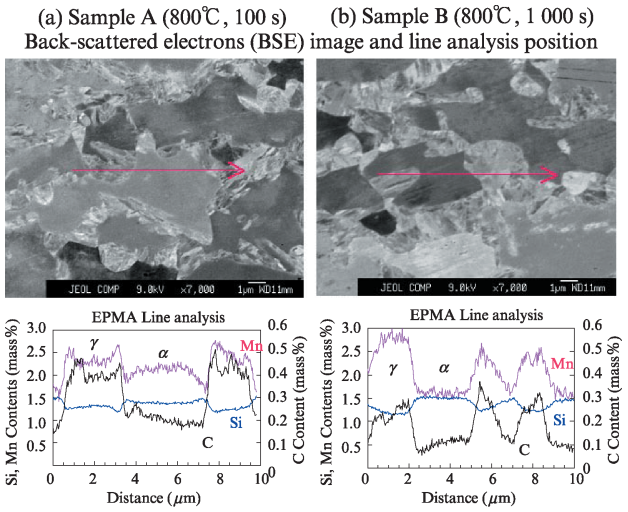

Fig. 4 Field emission-electron probe micro analyzer (FE-EPMA) quantitative line analysis of Si, Mn

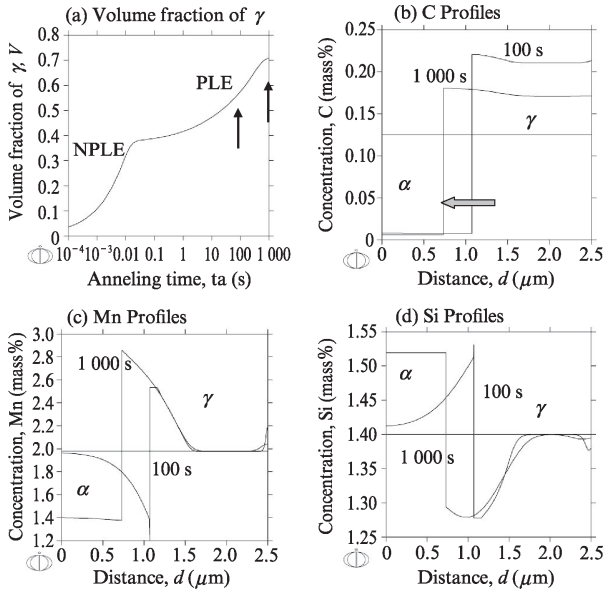

Fig. 5 (a) Calculated variation of the austenite volume fraction with annealing time and (b) C, (c) Mn, (d) Si profiles in 100 s, 1 000 s during 800˚C holding [8)]

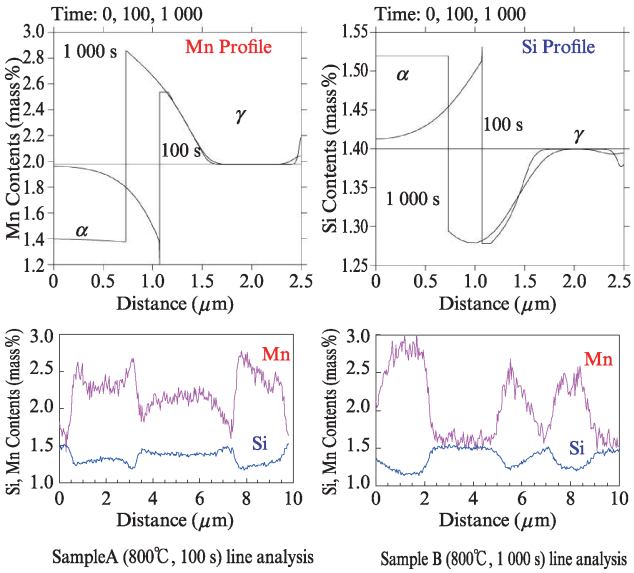

Fig. 6 Comparison between DICTRA Si, Mn profiles and field emission-electron probe micro analyzer (FE-EPMA) line analysis

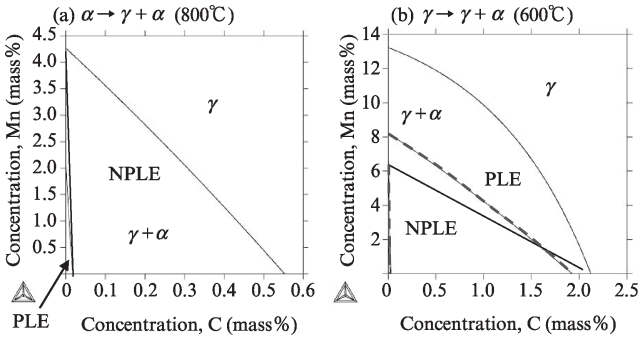

Fig. 7 Calculated isothermal section of Fe-1.4 mass%Si-Mn-C phase diagram at (a) 800˚C and (b) 600˚C [8)]

in both the α phase and γ phase, the amounts of Si and Mn in the center of the phase are almost the same as the steel composition, and diffusion has occurred only in the vicinity of the interface. On the other hand, in specimen B, which had a longer intercritical annealing time, the concentration of Si in the γ phase has decreased in comparison with specimen A, while the concentration of Mn in the γ phase has increased. Thus, distribution of both of the elements Si and Mn had progressed.

DICTRA calculations were performed in order to discuss the phase transformation behavior described above. The DICTRA calculation results of the isothermal transformation at 800˚C are shown in **Fig. 5**. Although this figure shows the change over time in the volume fraction of γ phase (logarithmic scale on x-axis) and the Si and Mn profiles in the cell after 0 s, 100 s and 1 000 s, it can be understood that the γ phase has grown from the boundary on the right side of the α single phase, and distribution of Si and Mn has occurred accompanying this. **Figure 6** shows a comparison of these profiles and the results of the above-mentioned FE-EPMA line analysis. The DICTRA calculations show good agreement with the line analysis results, as changes in the concentrations of both Si and Mn have occurred only at the interface after intercritical annealing for 100 s, and distribution has progressed after 1 000 s, and the concentrations of both Si and Mn in the α phase have become uniform.

Here, **Fig. 7** shows the isothermal section phase diagrams at 800˚C and 600˚C calculated by Thermo-Calc, and the PLE (Partition local equilibrium)/NPLE (Non-partition local equilibrium) boundary line of the thermal history of the cold-rolled DP steel in this research. Because intercritical annealing of the cold-rolled steel sheet at 800˚C results in an α+γ phase reverse transfor-

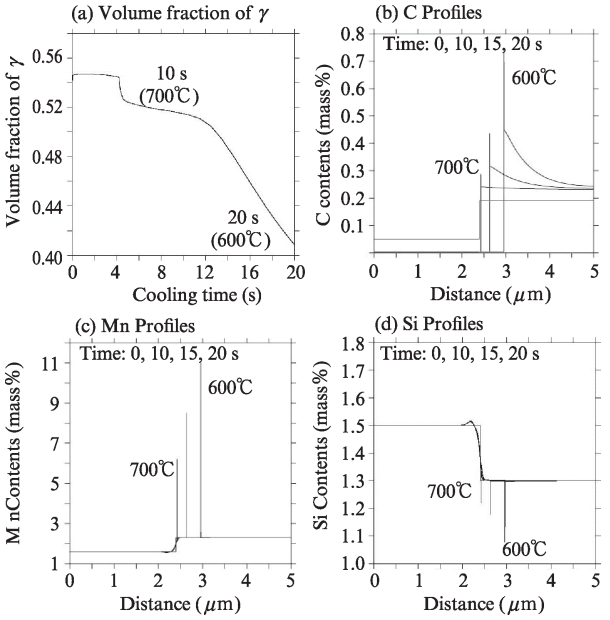

Fig. 8 (a) Calculated variation of the austenite volume fraction with cooling time from 800˚C, 100 s and (b) C, (c) Mn, (d) Si profiles [8)]

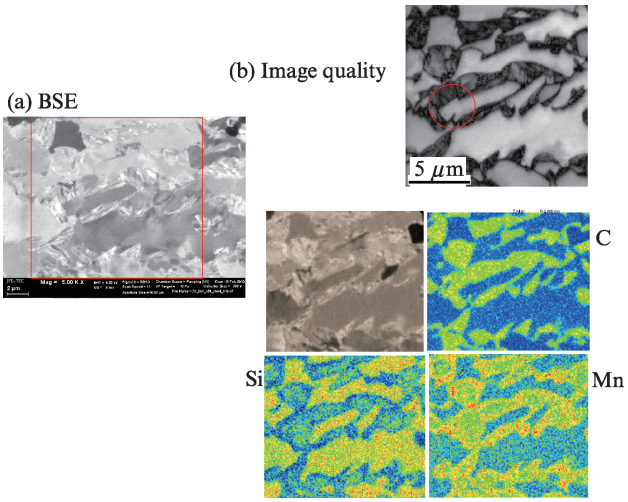

Fig. 9 (a) Back-scattered electrons (BSE) image, (b) Image quality of electron back-scattering pattern (EBSP) and (c) Field emission-electron probe micro analyzer (FE-EPMA) C, Mn, Si mapping on same position of sample C [8)]

mation from the α phase, the boundary line takes a vertical shape on the low C side, as shown in Fig. 7 (a). Accordingly, in the 800˚C isothermal transformation of the steel composition in this experiment, it is thought that the steel enters the PLE region from the NPLE region from the initial period of transformation due to distribution of C, and the transformation then proceeds in the PLE mode accompanying the diffusion of Si and Mn.

Because this kind of NPLE/PLE change is considered in the DICTRA calculations, the material enters the PLE region and distribution of Si and Mn occurs after 100 s and 1 000 s during intercritical annealing at 800˚C, as shown in Fig. 5 (a). As demonstrated by these results, analysis of the distribution behaviors of alloying elements such as Si, Mn, etc. by DICTRA is effective for evaluation of these dual phase steels.

Figure 8 shows the results of calculations of the change in the volume fraction of the γ phase and the C, Si and Mn profiles in specimen C, which was obtained by cooling specimen A (100 s) to 600˚C. These results are shown after times of 0,10 and 20 s, that is, at the temperatures of 800, 700 and 600˚C. From the figure, it can be understood that the $\gamma \rightarrow \alpha$ transformation progresses with cooling to 600˚C, and as a result, the volume fraction of γ phase decreases. As changes in the element profiles, these results reveal that C undergoes distribution with the movement of the interface, and the positions of the interfacial plane and changes in concentration coincide as C moves, but there is virtually no movement of Si and Mn, and only the spike moves. In other words, as shown in Fig. 7 (b), cooling from 800˚C to 600˚C proceeds under a NPLE or para-equilibrium condition. The results of FE-EPMA elemental mapping of specimen C are shown in **Fig. 9**. As shown in this figure, only the interface of the phase transformation moves, and there was an interface where distribution of Si and Mn did not occur (for example, at the position indicated by the red circle in Fig. 9 (b)).

4. Conclusion

The element distribution behavior of the α/martensite phase due to the dual phase microstructure of an Fe-C-Si-Mn quaternary model alloy was analyzed by FE-EPMA, together with calculations by DICTRA, which is a phase transformation analysis program for diffusion-controlled transformation. As a result, the following points were clarified.

(1) In isothermal annealing at 800˚C, distribution of C occurs rapidly from the initial stage of transformation, and as the transformation mode proceeds rapidly in the PLE (Partition local equilibrium) region.

(2) Accompanying this, the distribution of Si and Mn changes depending on the annealing time. The results of measurements of the distribution of Si and Mn by FE-EPMA were in good agreement with the calculated results.

(3) In continuous cooling to 600˚C after annealing at 800˚C, no changes could be seen in Si and Mn, and distribution of these elements did not occur. Because distribution of C proceeded, it is thought that the transformation during cooling proceeded under an NPLE (Non-partition local equilibrium) or para-equilibrium condition.

(4) Based on these results, in cold-rolled DP steel, the

distribution of the elements Si and Mn is decided by the holding time in isothermal annealing prior to cooling, indicating that the conditions of isothermal annealing are critical for determining the properties of the steel.

As demonstrated by this work, measurement of Si, Mn and other alloying elements by FE-EPMA and analysis by DICTRA is effective for evaluation of dual phase steels. JFE Steel is utilizing these analytical techniques in the development of new high tensile strength steels.

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