Distribution behavior of C and Mn among the matrix phase and martensite-austenite constituent in the heat-affected zone of a low alloy carbon steel

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Abstract: The present study investigated distribution behavior of C and Mn among the matrix phase and martensite-austenite constituent (MA) formed along laths in the simulated intercritically reheated coarse-grained HAZ (ICCGHAZ) thermal cycle of a low alloy carbon steel by Atom Probe Tomography (APT). In addition, correlation between the distribution of alloy elements and decomposition behavior of γ during intercritically reheated cycle was discussed. It was confirmed that MA formed along laths tended to remain after cooling at a rate of 1°C/s. Mn enriched in MA, which was originally austenite (γ) phase at higher temperature in the thermal cycle, in the vicinity of MA/ferrite(α) interface after intercritical annealing. Furthermore, distribution of Mn occurred in the vicinity of MA/matrix interface over 550°C during cooling at a rate of 10°C/s. Distribution of C occurred during cooling at a rate of 10°C/s from 750°C to room temperature. It is suggested that the transformation to α from γ formed along laths proceeds under the partition local equilibrium (PLE) during cooling over 550°C, therefore the MA formed along laths tended to remain after intercritically reheated cycle.

1. INTRODUCTION

The heat affected zone (HAZ) of steel formed during welding shows poor toughness due to the formation of martensite-austenite constituent (MA). In particular in the intercritically reheated coarse-grained HAZ (ICCGHAZ), it is known that C enrichment in austenite (γ) proceeds during intercritically reheated cycle and the formation of MA is promoted. Various studies about the macroscopic distribution behavior of alloy elements among MA and matrix have been made [1-3]. In the previous study using electron probe micro analyzer, it has been reported that C enriches in MA during short time annealing and fast cooling [3]. Furthermore, it has been concluded that Mn does not distribute between MA and the matrix phases. However, few studies have focused on distribution behavior in the vicinity of the MA/matrix interface during a short-time HAZ thermal cycle. In the present study, authors investigated the diffusion and distribution behaviors during the thermal cycle simulating ICCGHAZ by means of Atom Probe Tomography (APT), then distribution behavior of alloy elements among untransformed γ/matrix interface during intercritically reheated cycle was estimated. In addition, correlation between the distribution of alloy elements and decomposition behavior of γ was discussed.

2. EXPERIMENTAL PROCEDURE

Fe-0.054C-0.3Si-1.50Mn-0.032Al-0.050Nb (mass%) alloy was made by arc melting. Then, cylindrical specimens were cut out. Fig. 1 shows the thermal diagram. The specimens were held at 1400°C for 5s and cooled to room temperature (RT). Subsequently the specimens were reheated at 750°C for various periods to evaluate growth behavior of MA during intercritical annealing. After intercritical annealing, the specimens were cooled to RT at a rate of 10°C/s (slow cooling) or quenched into water at 750°C (750W.Q.) or 550°C (550W.Q.). APT analysis was carried out using speimens held at 750°C for 5s (750°C×5s) with various cooling conditions. In addition, a 750°C×5s-specimen was cooled at a rate of 1°C/s to compare decomposition behavior of γ during cooling process. The microstructures were etched with Lepera solution before optical microscope (OM) observation. MA for APT analysis were observed by EBSD to confirm that MA was whether martensite or γ phase, then martensite/matrix interfaces were cut out using foused ion beam. APT measurements were performed using LEAP 3000HR at a specimen temperature of -193°C (80K) and a pulse fraction of 15%.

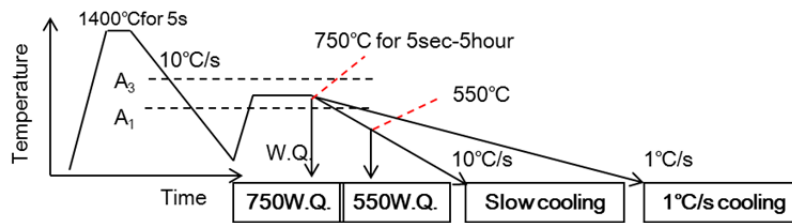* Corresponding author. E-mail: inomoto.masahiro@kobelco.com, telephone: +81-78-992-5503

Fig.1 Schematic thermal diagram

3. RESULTS AND DISUCUSSION

3.1. Microstructure

The OM images of the representative microstructure are shown in Fig.2 (a-e). MA particles are observed along laths and prior γ boundaries. Fig.3 shows relationship between holding time at 750°C and volume fraction of MA. The volume fraction of MA increases with an increase in holding time at 750°C. Lowering of cooling rate decreases the amount of MA. Although MA formed along laths becomes smaller with decreasing cooling rate as shown in Fig.2(a-c), it can be seen that MA formed along laths tends to retain after slow cooling and even cooling at a rate of 1°C/s. In contrast, it seems that MA formed along the prior γ boundaries easily decomposed to α+θ.
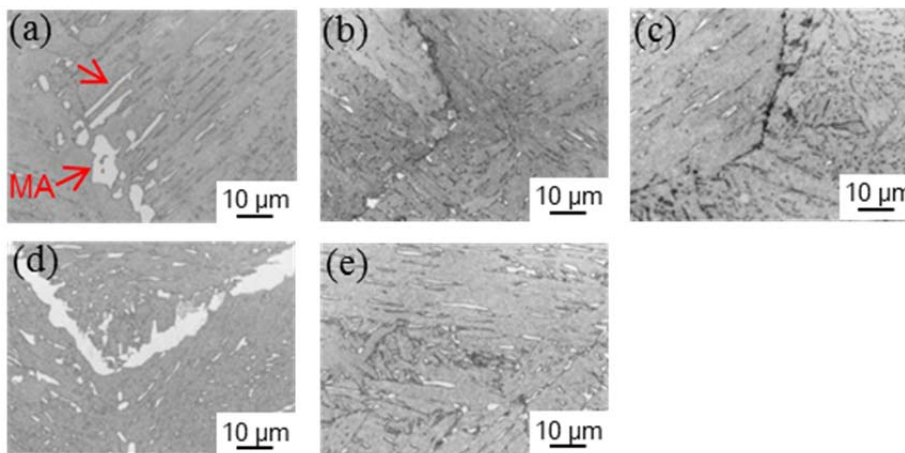

Fig.2 Optical micrographs etched by LePera solution. Conditions of intercritically reheated cycle are (a) 750°C×5s -750W.Q., (b) 750°C×5s-slow cooling, (c) 750°C×5s-1°C /s cooling, (d) 750°C×5h -750W.Q. and (e) 750°C×5h-slow cooling respectively.

3.2. APT Analysis

In order to assess decomposition behavior of γ along laths during intercritically reheated cycle, distribution of alloy elements among γ/matrix were estimated using alloy elements profiles measured by APT across the MA(martensite phase)/matrix interface.

Fig.4 (a, b) shows C and Mn profiles across the martensite/matrix interface in 750°C×5s-750W.Q., 750°C×5s-550W.Q. and 750°C×5s-slow cooling specimens. C concentrations in the martensite of 750°C×5s-750W.Q. and 750°C×5s-550W.Q. specimens are almost the same. Assuming that C atoms distribute during slow cooling from 750 to 550°C, it is considered that distribution of C among γ/matrix occurred during W.Q. from 750 to 550°C due to the rapid diffusion of C. By comparing C profiles of 750°C×5s-550W.Q. and 750°C×5s-slow cooling specimens, it can be seen that C enriched in γ during cooling from 550°C to RT.
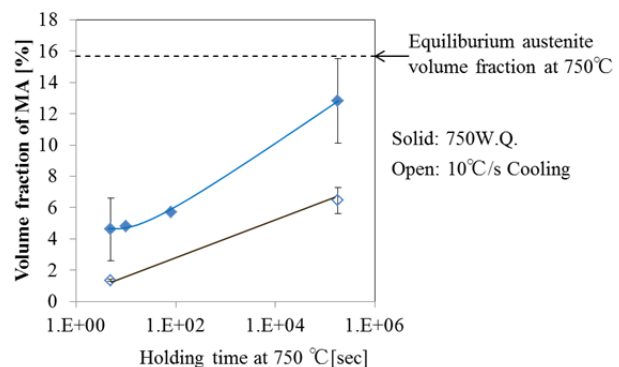

Fig.3 Relationship between holding time at 750°C and volume fraction of MA.
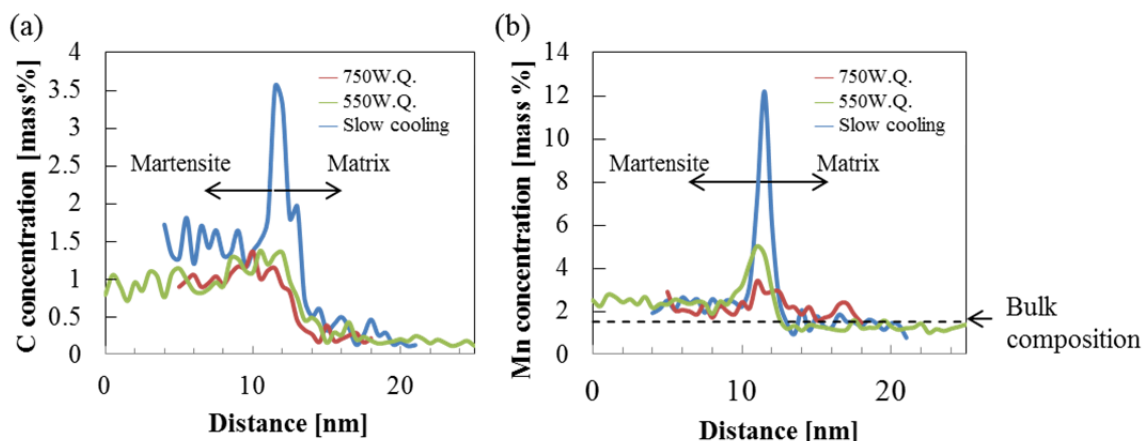
Fig.4 (a) C and (b) Mn profiles across the martensite/matrix interface of 750°C×5s-750W.Q., 750°C×5s-550W.Q. and 750°C×5s-slow cooling specimens [4].

Mn concentration in the matrix at 750°C is expected to be approximately the same as or less than bulk concentration. Nevertheless concentrations of Mn in the matrix and martensite of 750°C×5s-750W.Q. specimen were higher than the bulk concentration. This implies the matrix in the analysed volume of 750°C×5s-750W.Q. specimen at RT was considered to be originally γ at 750°C. It is presumed that the γ/matrix interface moved during W.Q. process, then diffusion of Mn could not catch up the movement of interface as shown in Fig.5. Furthermore, this indicates that Mn enrichment in γ occurred around the γ/matrix interface during intercritical annealing at 750°C even for 5s. As for slow cooling, it is also deduced that γ/α interface moved during slow cooling judging from the obsearavation in Fig.2 (a, b) that MA formed along laths in 750°C×5s-slow cooling specimen is smaller than that of 750°C×5s-750W.Q. one. In contrast, difference of Mn concentrations between MA and matrix is observed in 750°C×5s-550W.Q. and 750°C×5s-slow cooling specimens. In the slow cooling, it is considered that interface moving was accompanied by Mn diffusion.

Table 1 shows average concentration of Mn in martensite of each specimen. Mn concentration in the martensite of 750°C×5s-750W.Q. specimen is lower than that of 750°C×5s-550W.Q. and 750°C×5s-slow cooling ones. Furthermore, Mn concentrations in the martensite of 750°C×5s-550W.Q. and 750°C×5s-slow cooling specimens are almost the same. It can be found that distribution of Mn occurred during cooling at a rate of 10°C/s over 550°C. However, Mn distribution didn't occur under 550°C, then Mn segregation increased as shown in Fig.4 (b).

Table 1 Average concentration of Mn in martensite (mass%)

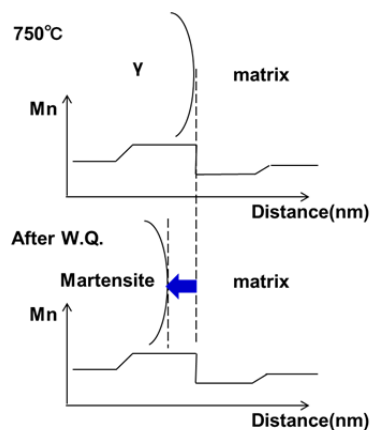| Heat treatment	Mn concentration (mass%)
750×5s-750W.Q.	2.12
750×5s-550W.Q.	2.39
750×5s-Slow cooling	2.31

Fig.5 Schemaic illustration of interface motion during W.Q. from 750°C.

3.3.α transformation from γ formed along laths during cooling

It is known that γ→α transformation in the Fe-C-M (M: substitutional alloy element) can proceeds without macroscopic diffusion of substitutional alloy elements in the case of fast cooling[5] because a diffusion rate of substitutional element is extremely lower than that of C. In order to evaluate α transformation behavior from γ formed along laths during cooling at a rate of 10°C /s from 750 to 550°C, NPLE (Non partition local equilibrium)/PLE (Partition local equilibrium) region was calculated by Thermo-Calc® software with the database of TCFE7. The bulk composition is at the

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NPLE region according to the calculation. However, APT analysis of 750×5s-750W.Q. specimen showed Mn enrichment in γ occurred in the vicinity of γ/α interface during intercritical annealing. Hence the γ→α transformation mode was considered based on the average composition of γ(Fe-2.1Mn-0.3Si-C) predicted by APT analysis of MA in 750°C×5s-750W.Q. specimen. Although the accurate C concentration in γ during cooling from 750 to 550°C was unclear, C concentration in MA of 750°C×5s-550W.Q. specimen was obviously higher than NPLE line as shown in Fig.6. Therefore it is presumed that transformation to α from γ formed along laths during cooling from 750 to 550°C proceeds under PLE mode by taking into account the C concentration in MA of 750°C×5s-550W.Q. specimen and slight distribution of the substitutional element (Mn). This estimation corresponds to the result that MA formed along laths remained even after cooling at a rate of 1°C/s. In other words, decomposition of MA along laths do not likely occur with cooling process, since the γ formed along laths transforms slowly to α due to PLE mode.
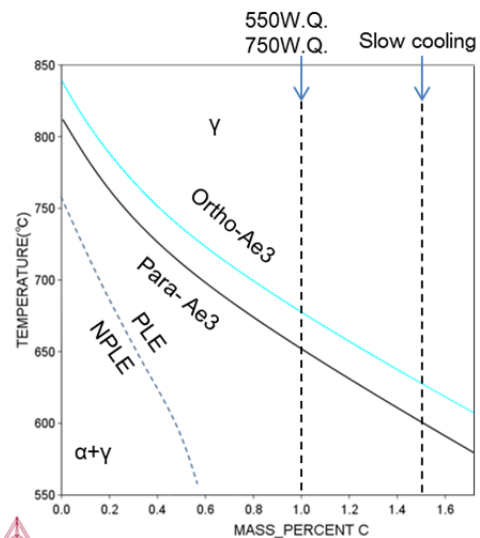

Fig.6 Average C cocentration in martensite analyzed using APT superimposed on the phase diagram of Fe-0.3Si-2.1Mn-C alloy.

4. CONCLUSION

Distribution behavior of C and Mn among the matrix phase and MA formed along laths in the ICCGHAZ of a low alloy carbon steel was investigated using APT. In addition, correlation between the distribution of alloy elements and decomposition behavior of γ during intercritically reheated cycle was discussed.

MA formed along laths tended to remain after cooling at a rate of 1°C/s. APT analysis revealed that Mn enriched in MA, which was originally γ phase at higher temperature in the thermal cycle, in the vicinity of MA/matrix interface after intercritical annealing. In addition, distribution of Mn occurred in the vicinity of MA/matirix interface over 550°C during cooling at a rate of 10°C/s. Distribution of C occurred during cooling at a rate of 10°C/s from 750°C to room temperature. It is suggested that the transformation to α from γ formed along laths proceeds under the partition local equilibrium (PLE) during cooling over 550°C, therefore the MA formed along laths tended to remain after intercritically reheated cycle.

REFERENCES

[1]   H. Okada, K. Ikeuchi, F. Matsuda, I. Hrivnak, Z. Li , Quarterly J. Japan Weld. Soc., 12-2 (1994), 236-242.
[2]   Y. Komizo, Y. Fukada, Quarterly J. Japan Weld. Soc., 6-1 (1988), 41-46.
[3]   S. Endo, Tetsu-to-Hagané, 84 (1998), 297-302.
[4]   M. Inomoto, H. Nako, CAMP-ISIJ, 30 (2017), 434.
[5]   M. Enomoto, Materia Japan, 54 (2015), 12-17.