Note

Transition from Diffusive to Displacive Austenite Reversion in Low-Alloy Steel

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For understanding the transition from diffusive to displacive austenite reversion mechanism in steel, the effect of heating rate on austenite reversion behavior was investigated in 0.15%C–5%Mn steel. Austenite reversion temperature first increased gradually with the heating rate owing to the superheating effect and then remained at a constant temperature above a critical heating rate. In response, the austenite formed by rapid heating exhibited a coarse prior austenite grain structure, indicating the occurrence of displacive reversion even in low-alloy steel.

KEY WORDS: displacive reversion; transition of reversion mechanism; low-alloy steel; heating rate.

Martensitic transformation caused by diffusionless shear takes place athermally in steel when the decomposition of austenite to such products as ferrite, pearlite and bainite is suppressed during cooling. In general, martensitic transformation depends primarily on the cooling rate and composition of steel. A high cooling rate and/or the addition of alloying elements to avoid diffusive reactions results in a significant degree of super cooling. Eventually, the large driving force necessary for martensitic transformation (about 1200 J/mol) can be obtained at the martensite-start temperature, M_s . In principle, martensitic transformation is reversible. It is known that the reversible transformation from martensite to austenite (martensitic reversion) occurs in maraging steel owing to its high Ni content.^{1–4)} Previously,⁵⁾ the authors of the current study directly observed an austenitic structure formed by martensitic reversion (martensitically reversed austenite) in 18%Ni–C (% = wt.%) steel. It was reported that after reheating the sample to a temperature above the martensitic austenite-start temperature, A_s , all martensite laths originally formed via an fcc→bcc martensitic transformation reverse into new austenite during the bcc→fcc martensitic reversion, thus returning to an original austenitic structure with the same crystallographic orientation. However, the mechanism of this two-directional transformation is still under debate because such martensitic reversion almost never occurs in low-alloyed steels. The main reason that inhibits such reversibility is the high A_s peculiar to athermal martensitic transformation.

Martensite decomposition (*i.e.*, diffusive reversion and cementite precipitation) occurs easily during reheating toward A_s because diffusion is easy in low-alloyed steel at elevated temperatures around A_s . This difference in the reversion mechanism between high Ni or Mn steel (*e.g.* 18% Ni steel) and low-alloyed steel is schematically shown in Fig. 1 in the form of a continuous heating reversion diagram. In these diagrams, the austenite-start and -finish temperatures for diffusive reversion during heating are defined as Ac_1 and Ac_3 , respectively, for distinguishing them from the austenite-start and -finish temperatures for martensitic reversion, A_s and A_f . Although diffusive reversion is preferential, upon observing Fig. 1(b), one can expect that the reversion mechanism is changeable depending on the heating rate and that martensitic reversion should occur even in low-alloyed steels if the heating rate is sufficient for avoiding the diffusive reversion. In this study, the effect of heating rate on reversion behavior was investigated in 0.15%C–5%Mn steel for understanding the reversion transition mechanism in steel.

0.15%C–5%Mn steel (0.14%C–4.97%Mn; % = wt.%), was used in this study as a low-carbon and low-alloyed steel†. Mn addition lowers T_0 (T_0 : temperature where the Gibbs free energies of fcc and bcc are equal) as well as atomic diffusivity in steel, which is thought to promote the occurrence of martensitic reversion. The material was initially solution treated at 1273 K in the austenite single-phase region for 1.8 ks, followed by water quenching to obtain an initial lath martensitic structure. Subsequently, this solution-treated material was cut into cylindrical rods of length 10 mm and diameter 2 mm; these rods were re-austenitized at different heating rates from 20 to 450 K/s using induction heating apparatus. The transformation temperature during re-austenitization was determined using a thermal dilatation tester equipped with a temperature control system. In this system, temperature is measured by an extra-fine thermocouple having high-speed thermal response. Dislocation density in the martensite was calculated by the X-ray diffraction method using Co-K α radiation according to Hall-Williamson's method.^{6,7)} The microstructure and crystallographic characteristics of the rods were analyzed by the electron back-scattering diffraction (EBSD) method using a field-emission scanning electron microscope (JSM-6701F developed by JEOL Ltd.).

For visualizing prior austenite grain boundaries in the lath martensitic structure, a specific technique was used in the crystallographic analysis. In general, each martensite variant has a Kurdjumov–Sachs (K–S) orientation relationship with prior austenite in low-carbon steels. Therefore, block and packet boundaries should lie between different two of 24 K–S

Fig. 1. Schematic diagram of continuous heating transformation explaining the difference in reversion mechanism between high Ni or Mn steel (a) and low-alloy steel (b).

† The definition to distinguish 'low' and 'high' alloy steels varies among countries and among standard-setting organizations. As a general indication, low-alloy steel can be regarded as alloy steels containing 1–5% of elements except carbon by the ISO definition.

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Fig. 2. Distribution of misorientation angle in lath martensitic structure and random orientation structure.

variants and should have specific grain boundary characteristics. The possible misorientation angle characterized by two different K–S variants can be calculated geometrically, as reported by Morito *et al.*⁸⁾ Actual distribution of the misorientation angle in the lath martensitic structure of 0.15%C–5%Mn steel (shown later in Fig. 4(a)) is shown in Fig. 2. Given that a block is a smaller subunit than a packet, the density of block boundaries is considerably higher than that of packet boundaries. Therefore, the distribution profile has clear peaks with deviations of several degrees at approximately 10.5°, 49.5° and 60°, which are the theoretical misorientation angles of block boundaries in a K–S relationship. By contrast, prior austenite grain boundaries should have a random misorientation distribution, *i.e.*, a Mackenzie profile,⁹⁾ because the two martensite variants that form a prior austenite grain boundary had been in different prior austenite grains and, therefore, no orientation relationship. That is, to observe prior austenite grain boundaries, we should focus on the difference between the specific misorientation distribution in the lath martensitic structure and the Mackenzie profile shown in Fig. 2. When we selected the misorientation selection window between 20 to 50° in the orientation analysis software, the fraction of the grain boundary in the selection window was 72% in the Mackenzie profile. This means that the selection window enables us to visualize the majority of prior austenite grain boundaries, excluding block and packet boundaries in the lath martensitic structure.

Figure 3 shows the continuous heating reversion diagram of 0.15%C–5%Mn steel, including changes in the austenite-start and -finish temperatures as a function of the heating rate. Additionally, A_{e1} (780 K), A_{e3} (997 K) and T_0 (883 K), as calculated by *Thermo-calc.* (SSOL6 database), are also shown in this diagram. Because A_{e1} was too low for promoting atomic diffusion of iron, there is a large gap between A_{e1} and the austenite-start temperature even at the slowest heating rate. However, the austenite-finish temperature was almost the same as A_{e3} under the heating rate of 20 K/s, which suggests that diffusive reversion must have been completed under near thermal equilibrium conditions in the case of slow heating. The austenite-start and -finish temperatures increased gradually with the heating rate, and the austenite-finish temperature was higher than A_{e3} by 50 K under the heating rate of 300 K/s. The austenite-finish temperature increased along the dashed line, which is Ac_3 , as roughly estimated using *DICTRA* (Diffusion Controlled Transformation, SSOL6 and Mob2 databases). This implies that the heating rate dependence of the reversion temperature was due to the superheating effect, and diffusive reversion took place at heating rates of up to 300 K/s. According to the estimated Ac_3 , the reversion should have been completed at

Fig. 3. Continuous heating reversion diagram in 0.15%C–5%Mn steel.

1 130 K under the heating rate of 400 K/s. However, the austenite-finish temperature was much lower in practice, and the austenite-start and -finish temperatures remained constant at 1006 ± 8 and 1047 ± 6 K, respectively, under further rapid heating. Such discontinuous changes in the reversion temperature correspond to Fig. 1(b), that is, this result implies that the transformation mechanism changes from diffusive to martensitic reversion as the heating rate is increased. Some researchers investigated martensitic reversion in some types of high-alloyed steels,^{10,11)} and the driving force necessary for martensitic reversion was calculated to be 300–400 J/mol from its own As . *Thermo-calc.* results indicate that the Gibbs free energy gap between fcc and bcc in this material is approximately –350 J/mol at 1 000 K if carbide does not precipitate within the lath martensite matrix during heating, which allows the occurrence of martensitic reversion in terms of thermodynamics. However, we cannot ignore carbon diffusion at such elevated temperatures. Assuming that a transformation interface migrates 10 μm , which corresponds to a conventional martensite block size during heating from Ac_1 to Ac_3 , the length of carbon diffusion field in the martensite matrix at the austenite/martensite interface, L , can be estimated as being approximately 1 μm from the diffusion coefficient of carbon, D , and the interface velocity, v , *i.e.*, $L \sim D/v$.¹²⁾ This estimate suggests that even if martensite reverses to austenite via a displacive mechanism, the austenite reversion should be accompanied by carbon diffusion.

Figure 4 shows the bcc orientation imaging maps (Figs. 4(a-1)–4(d-1)) and the corresponding boundary maps with misorientation angles over 15° (Figs. 4(a-2)–4(d-2)) and 20–50° (Figs. 4(a-3)–4(d-3)) obtained by high-resolution EBSD at a step size of 500 nm. The maps show the microstructure of the solution-treated specimens and the re-austenitized specimens reheated at heating rates of 200, 300 and 450 K/s. Each re-austenitized specimen was reheated at the respective heating rate and then held for 10 s at 1 073 K, which is higher than the austenite-finish temperatures (see Fig. 3), followed by He gas quenching. All specimens had a typical lath martensitic structure consisting of packet and block boundaries (Figs. 4(a-1)–4(d-1)), and X-ray diffraction analysis proved there was no retained austenite. Boundary maps with misorientation angles over 15° (Figs. 4(a-2)–4(d-2)) indicate that the lath martensitic structure is characterized by high angle block and packet boundaries and that there is no significant difference in the density of these boundaries among specimens. To reveal the occurrence of the displacive reversion, we must focus not on block and packet boundaries but on the prior austenite grain boundaries; this is because while the original austenite grain boundaries remained in displacive reversion,^{3,5)} new austenite grains

Fig. 4. Orientation imaging map and grain boundary map of 0.15%C–5%Mn steel. Initial solution treated material (a) and re-austenitized materials at heating rate of 200 (b), 300 (c) and 450 K/s (d).

were formed through nucleation and growth in diffusive reversion. In other words, the austenite grain size does not change during displacive transformations. Therefore, boundary maps with misorientation angles of 20–50° were shown in this figure for visualizing the prior austenite grain structure. The shape of the prior austenite grains in the solution-treated material can be observed roughly in Fig. 4(a-3), and it was found that the initial austenite grain size was approximately 100 μm . The prior austenite grains were significantly refined by re-austenitization at heating rates of 200 and 300 K/s, and the grain size decreased slightly up on increasing the heating rate (Figs. 4(b-3), 4(c-3)). This implies that diffusive reversion occurred under these heating conditions and that the superheating effect should enhance the austenite nucleation frequency. It should be noted that the austenite formed under the heating rate of 450 K/s had coarse prior austenite grains, not the fine ones (Fig. 4(d-3)). Indeed, the grain boundary length per unit area of each specimen was evaluated at (a-3) 5.9×10^4 , (b-3) 18.3×10^4 , (c-3) 20.5×10^4 and (d-3) 13.2×10^4 m/m^2 . The presence of a coarse-grained prior austenitic structure demonstrates that the reversion mechanism was changed discontinuously by rapid heating above a critical heating rate (between 300 and 400 K/s). Furthermore, it was confirmed that there is little difference in the hardness and dislocation density among the solution-treated specimens and the re-austenitized specimens reheated under heating rates of 200 and 300 K/s [4.00 ± 0.03 GPa, $(3.7 \pm 0.1) \times 10^{15}$ m^{-2}], but the re-austenitized specimen reheated at a heating rate of 450 K/s had higher hardness and dislocation density (4.48 GPa, 4.6×10^{15} m^{-2}). These results strongly suggest that the austenite formed by displacive reversion exhibits a high dislocation density, which contributes to the strengthening and toughening of martensite after quenching due to an ausforming-like effect.^{4,13,14} In contrast, a few fine austenite grains were found to be dispersed within the coarse-grained matrix (Fig. 4(d-3)), as indicated by the arrows. It is known that martensitically reversed austenite is easy to recrystallize at elevated temperatures owing to a high dislocation density.^{1–5,15} Therefore, it is thought that displacive reversed austenite statically recrystallizes in a short time resulting in the formation of fine austenite grains. These experimental results demonstrate that displacive reversion accompanied by carbon diffusion takes place even in low-alloyed steels when the heating rate is sufficient for suppressing diffusive reversion and also that the displacive-reversed austenite can easily recrystallize.

For understanding the reversion mechanism in steel, the

effect of the heating rate on austenite reversion behavior was investigated in 0.15%C–5%Mn steel. The results obtained are summarized as follows:

- (1) Displacive reversion takes place even in low-alloy steels when the heating rate is above a critical value for avoiding diffusive reversion.
- (2) In diffusive reversion, the reversion temperature increases gradually with the heating rate owing to the superheating effect, but remains constant in displacive reversion.
- (3) Specimens re-austenitized through displacive reversion had a lath martensitic structure with coarse prior austenite grains, and higher hardness and dislocation density than the specimen before re-austenitization.

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