Microstructure Evolution during Reverse Transformation of Austenite from Tempered Martensite in Low Alloy Steel

Tomoya SHINOZAKI,1)* Yo TOMOTA,2) Tatsuya FUKINO3) and Tetsuya SUZUKI4)

1) Steel Casting and Forging Division, Kobe Steel, Ltd., 2-3-1 Shinhama Arai-cho, Takasago, Hyogo, 676-8670 Japan.
2) National Institute for Materials Science, 1-2-1 Sengen, Tsukuba, Ibaraki, 305-0047 Japan.
3) TSL Solutions, Ltd., SIC2-401, 5-4-30 Nishihashimoto, Midori-ku, Sagamihara, Kanagawa, 252-0131 Japan.
4) Department of Materials Science and Engineering, Ibaraki University, 4-12-1 Nakanarusawa, Hitachi, Ibaraki, 316-8511 Japan.

* Corresponding author: E-mail: shinozaki.tomoya@kobelco.com
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Microstructure evolution during the reverse transformation of low alloy steel consisting of lath martensite and chromium carbide was examined using in-situ electron backscatter diffraction at high temperatures. Austenite grains nucleated during the reverse transformation were categorized into two types: austenite (type A), and austenite grains nucleated at the prior austenite grain boundaries or inside the prior austenite grains with a different crystal orientation (type B). After the reverse transformation was finished, the prior austenite grains were reconstructed by the rapid growth and coalescence of type-A grains, which was called the “austenite memory phenomenon.” Here, misorientation remained in the reconstructed austenite grains. Hence, upon heating to a higher temperature, type-A grains were invaded and were eventually replaced by type-B grains, resulting in a new fine-grained microstructure; this was similar to recrystallization. Therefore, these results showed that the austenite memory phenomenon occurred when the nucleation and growth of type-A grains was more dominant than those of type-B grains and that the degree of grain refinement depended on the nucleation and growth rate of the type-B grains.

KEY WORDS: reverse transformation; austenite memory; tempered martensite; recrystallization; variant selection.

1. Introduction

Grain refinement is effective means of simultaneously improving strength and toughness. Phase transformation from ferrite (α) to austenite (γ) (hereinafter referred to as “reverse transformation”) is often used to refine the γ grain size in the steel production process. However, the degree of grain refinement depends on the heating rate1–3) and initial microstructure.3,5) When martensitic and/or bainitic steel is heated to an austenitizing temperature, the newly formed γ grains have the same size and shape as the prior γ grains in some cases. This is called “γ memory”6,7) or “abnormal reverse transformation”.7) Interestingly, the coarse γ grains formed immediately after the completion of reverse transformation are replaced by smaller γ grains at a much higher temperature than Ac3.7,9)

Numerous studies have already been conducted in an attempt to understand these phenomena. Regarding the formation of reversed γ from martensite or bainite, it has been reported that there are two types of reversed γ, i.e., “globular γ” and “acicular γ”.3,6,7,10–12) Globular γ grains nucleate mainly at the prior γ grain boundaries, whereas acicular γ grains, which have a fine needle-shaped morphology, nucleate and grow at the lath boundaries of martensite or bainite. It has been estimated that the acicular γ grains within certain prior γ grains have an identical crystal orientation and that the reconstitution of the prior γ grains is achieved by the growth, impingement, and coalescence of the acicular γ grains.

Several possible mechanisms for the formation of acicular γ, related to the γ memory phenomenon, have been proposed. These are classified into three groups: (1) martensitic reverse transformation,11,13–15) (2) variant restriction,16,17) and (3) the growth and coalescence of retained γ6,7) These are summarized as follows.

(1) Several researchers have proposed that acicular γ is formed by martensitic reverse transformation, which is a diffusionless shear mechanism. Many researchers11,13,15) have claimed that a Kurdjumov Sacks (K-S) crystal orientation relationship (OR) exists between the acicular γ and initial martensite matrix. It has been observed that the reversed γ has a fine lath structure with a high dislocation density.15) It has been assumed that the grain refining behavior after the completion of the reverse transformation occurs as a result of the recrystallization, with the high dislocation density as the driving force.6,17)

(2) Watanabe et al.18) reported that, for a steel con-
taining cementite, the reversed γ nucleated with a special crystal orientation relationship between the cementite, α, and γ phases. On the other hand, Nakada et al.\(^5\) showed that, in an ultralow carbon martensitic stainless steel with no cementite, the reversed γ selected exactly the same variant as the prior γ. They concluded that internal stress within the initial martensite controlled the variant selection of reversed γ.

(3) Kimmins et al.\(^5\) and Hara et al.\(^7\) suggested the possibility of the growth and coalescence of the retained γ. If a bainitic or martensitic structure contains the retained γ, each retained γ grain within a given prior γ grain would have the same crystal orientation. The retained γ grows and coalesces on impingement upon heating, resulting in the reconstitution of the original prior γ grain.

As described above, the possibility of diffusionless transformation has been discussed in previous studies. In this case, it is necessary to avoid diffusive reactions like those that occur when using iron-nickel alloys like maraging steel\(^14,15\) and/or a high heating rate\(^20\). Such conditions are not applicable to the reverse transformation occurring at a slow heating rate in low alloy steel, hence, other mechanisms should be discussed. The grain refining behavior that occurs after reverse transformation has been poorly investigated. One of the reasons for this is the fact that conventional microstructure observation techniques cannot reveal the characteristics of the γ phase in low alloy steel at elevated temperatures. If a significant amount of the untransformed γ could be retained at room temperature, as in the case of 18%Ni maraging steel, the reversed γ could be analyzed in detail. However, the amount of retained γ in low alloy steel is extremely low at room temperature.

Recently, heating stages for a scanning electron microscope (SEM)/electron backscatter diffraction (EBSD) technique have been developed to examine the microstructure change at elevated temperatures during heating.\(^21-26\) This in-situ SEM/EBSD technique would make it possible to directly observe the crystallographic features of γ grains. Therefore, in this study, the reverse transformation of γ from tempered martensite at a slow heating rate in a low alloy steel was investigated using the in-situ SEM/EBSD method to clarify the mechanism for γ memory and grain refining behavior.

2. Experimental Procedures

The steel used in this study was melted in a vacuum induction furnace and cast as 20 kg ingots. The chemical composition of the steel was 0.36C-0.22Si-0.79Mn-0.007P-0.003S-3.04Cr-1.46Ni-0.43Mo-0.1V-0.03Al (in mass%) and balanced Fe. The ingots were forged into 40 mm square billets, from which 20 mm cubic specimens were machined. The specimens were annealed at 1453 K for 3.6 ks, followed by quenching in water. The quenched specimens were tempered at 973 K for 54 ks aiming at the precipitation of carbides and the decomposition of the retained γ. The retained γ in the specimen after the above heat treatment was examined using X-ray diffraction and no retained γ was detected. Some specimens were reheated at various temperatures between 1013 and 1193 K at a heating rate of 0.01 K/s and then quenched in water to observe the traces of γ grains. The \(A_C1\) and \(A_C3\) temperatures determined by dilatometry were 1003 K and 1088 K, respectively.

Microstructure observations were carried out using an optical microscope (OM), a SEM and a transmission electron microscope (TEM). The specimens for the OM and SEM observations were mechanically polished and etched with 3% nital. The prior γ grain boundaries were revealed by etching with a solution of picric acid. The carbides were examined using electrolytically extracted residue and carbon extraction replica techniques. The residue was separated using a membrane filter with 0.1 μm pores. The extracted carbide powder was subjected to chemical analysis and X-ray diffraction (XRD) with a Cu target to clarify the type of carbides. The carbon replica was used in TEM observation. The chemical compositions of the carbides were measured using energy dispersive X-ray spectroscopy (EDX).

EBSD measurements during annealing were carried out using a field emission (FE)-SEM with a heating stage (TSL solutions Co., Ltd.; HSEA-1000). A plate specimen that was 57 × 7 × 0.7 (mm\(^3\)) in size was cut from the quenched and tempered specimens. In this experiment, the temperature was increased in steps to obtain EBSD patterns at various temperatures from 973 to 1173 K with a heating rate of 0.05 K/s, as illustrated in Fig. 1. The EBSD measurements were performed in a 400 μm square region with a step size of 1 μm during an isothermal holding of 2 ks. The obtained EBSD patterns were analyzed using an OIM system (EDAX Inc.; TSL OIM Analysis ver.7.2.1)

3. Results and Discussion

3.1. Initial Microstructure

Figure 2 shows OM and TEM images of a specimen subjected to the previously described heat treatment. The specimen has a typical tempered lath martensitic structure. Coarse prior γ grains with size exceeding 1 mm are observed. As can be seen in the TEM image (Fig. 2(b)), the martensitic lath structure still has been retained even though a significant recovery of the dislocation substructure has occurred because of the high temperature tempering. The retained γ is not observed in the TEM observation results. Meanwhile, precipitation particles are seen at the lath boundaries. The results of EDX analysis suggest that most of the particles can be identified as alloy carbide as shown in Fig. 2(c). To quantitatively clarify the type of precipitation, an extracted residue analysis was carried out.
Figure 3 shows the XRD profile of the extracted residue. The peaks indicated by circles in Fig. 3 correspond to $M_7C_3$ (chromium carbide) while those indicated by triangles are $M_3C$ (cementite). The peaks of the XRD profile were mainly chromium carbide although a tiny amount of cementite was detected. The phase fraction of cementite in Fig. 3 was approximately 4%.

### 3.2. Microstructure of Specimen Quenched after Austenitization at Slow Heating Rate

Figure 4 shows OM micrographs revealing the prior $\gamma$ grain boundary (PAGB) of specimens that were reheated to various temperatures between 1 103 and 1 193 K for 3.6 ks at a slow reheating rate of 0.01 K/s and then quenched in water. The specimen quenched after austenitization at 1 103 K has coarse grains with size exceeding 1 mm, as shown in Fig. 4(a), indicating no significant change in size compared to the prior $\gamma$ grains observed in Fig. 2(a). On the other hand, in the specimen austenitized at 1 163 K, some small grains are found to have been generated at the PAGB, as indicated by the arrows in Fig. 4(b). With a further increase in the austenitizing temperature, the number of small grains increases, resulting in a fine $\gamma$ grain structure, as shown in Fig. 4(c).

To acquire a greater insight into the reverse transformation behavior of the present steel, the formation process of the reversed $\gamma$ was examined using SEM. Figure 5 shows SEM images of specimens of the two-phase ($\alpha+\gamma$) region, quenched immediately after being reheated to 1 013 K and 1 048 K at a heating rate of 0.01 K/s. In the specimen quenched after being reheated to 1 013 K (which was just above the $\Lambda_C1$ temperature), needle-shaped (acicular) martensitic structures ($\alpha'$) are observed along the lath boundaries of the initial tempered martensite, as indicated by the white arrows in Fig. 5(a). The matrix shows the untransformed initial structure, i.e., tempered martensite, while the area of fresh martensite corresponds to that of the reversed $\gamma$. In the specimen quenched after being reheated to 1 048 K, a large amount of acicular grains are formed as shown in Fig. 5(b). Additionally, some precipitation particles are observed.
in those SEM images. Figure 6 shows the results of TEM observation of the carbon extracted replica on a specimen quenched after being reheated to 1 048 K. A large number of precipitation particles are observed, as can be seen in the TEM image shown in Fig. 6(a). Figure 6(c) shows the results of EDX analysis for three typical particles shown in Fig. 6(b). Most particles are identified as alloy carbides, which is almost the same as the carbides in a specimen before reheating, as indicated in Fig. 2(c). Consequently, alloy carbides of M\textsubscript{7}C\textsubscript{3}, which are very stable, are found undissolved during the reverse transformation. It is observed that the acicular grains grow and coalesce with an increase in the reheating temperature, leading to the reconstitution of coarse prior \(\gamma\) grains before the reverse transformation. However, there is a need to directly observe such a process in-situ at elevated temperatures.

3.3. In-situ SEM/EBSD Observation of Reverse Transformation during Annealing

Figures 7 and 8 show the results of in-situ SEM/EBSD measurements during annealing at several temperatures. Figure 5(a) shows inverse pole figure (IPF) maps of the \(\alpha\) phase obtained at 973 K. A fully tempered martensitic structure with no retained \(\gamma\) was observed at temperatures below \(\text{AC}_1\). When the specimen was heated to 1 048 K, the \(\gamma\) phase was detected by the EBSD measurement. Figures 7(b)–7(e) present the phase map (b) and IPF maps (c–e) of the \(\gamma\) phase obtained during annealing at 1 048 K. Figures 7(d) and 7(e) show magnified images of framed areas 1 and 2 in Fig. 7(c), respectively. The phase fraction of \(\gamma\) in Fig. 7(b) is approximately 51%, which is in good agreement with the results determined using dilatometry. As seen in Fig. 7(c), the reversed \(\gamma\) grains that nucleated along the lath boundaries have almost the same crystal orientation. Additionally, a small number of fine \(\gamma\) grains with different crystal orientations nucleated at the PAGB or inside the prior \(\gamma\) grains as indicated by white arrows in Figs. 7(d) and 7(e).

Figures 8(a) and 8(b) show IPF maps obtained at 1 123 K and 1 173 K, respectively. The reverse transformation had already been completed at temperatures below 1 123 K. As can be seen in Fig. 8(a), reversed \(\gamma\) grains with a specific crystal orientation were mainly produced, and eventually constituted a \(\gamma\) grain of the same size as the prior \(\gamma\) grains. Then, with further reheating to 1 173 K, some polygonal grains with different crystal orientations were produced and replaced the coarse grains. These behaviors are more obvious in the Kernel average misorientation (KAM) map. Figures 8(c) and (d) show KAM maps for \(\gamma\) obtained at 1 123 K and 1 173 K, respectively. It is found that there are large KAM values within the coarse \(\gamma\) grains, immediately after the completion of the reverse transformation, whereas the fine grains indicated by the white arrows in Fig. 8(c) have a low KAM value. The lower region with the low KAM value, shown in Fig. 8(d) corresponds to the newly apparent polygonal grains shown in Fig. 8(b), whereas the upper region with the high KAM value in Fig. 8(d) corresponds to the previously transformed grains, colored blue and violet in Figs. 8(a) and 8(b). An island region with a high KAM value in the middle of Fig. 8(d) was found to not appear newly but instead mixed with the former violet grains and invading red and blue polygonal grains. Some of the polygonal grains observed in Fig. 8(b) do not correspond to the tiny grains in Fig. 8(a) (see white arrows). Such new polygonal grains are thought to appear on the surface with the growth of those grains that have nucleated in the interior of the specimen.

Those grains with a low KAM value correspond to fine reversed \(\gamma\) grains with different crystal orientations, nucleated at the PAGB or inside the grains. When the specimen was reheated at 1 173 K, the coarse \(\gamma\) grains were gradually invaded and replaced by some grains with a low KAM value (see Fig. 8(d)). As a result, a new small grain structure was formed at a temperature higher than \(\text{AC}_3\).

After the EBSD measurements, the specimen was examined using an electron probe micro-analyzer (EPMA) on the surface and at a cross section. Although the Mn atoms near the surface had completely evaporated and the carbon concentration had slightly decreased, the influence of the change in the chemical composition during the in-situ EBSD measurements on the previously mentioned conclusions is negligible in the case of the present Ni–Cr–Mo steel.

3.4. Crystallographic Characteristics of Reversed Austenite

To understand the \(\gamma\) memory phenomenon, it was necessary to clarify the relationship between the reversed and prior \(\gamma\) grains. However, it was impossible to directly determine the crystal orientation of the prior \(\gamma\) grains because the \(\gamma\) was not retained in the initial tempered martensitic structure. Lath martensite is well known to show the K–S OR with respect to the prior \(\gamma\).\textsuperscript{27} Thus, it would be possible to estimate the crystal orientation of the prior \(\gamma\) from that of the martensite if the K–S OR held. Recently, a novel technique has been developed for reconstructing the prior \(\gamma\) orientation from the \(\gamma\) orientation using the SEM/EBSD data.\textsuperscript{28–31} Therefore, the prior \(\gamma\) grains were reconstructed.
Fig. 7. SEM/EBSD images obtained during annealing: (a) at 973 K and (b–e) at 1 048 K. (a) Inverse pole figure (IPF) map of ferrite. (b) Phase map where red and green refer to ferrite and \( \gamma \) respectively. (c–e) IPF maps of \( \gamma \) where magnified images (d) and (e) correspond to the square framed areas 1 and 2 in (c).

Fig. 8. SEM/EBSD images obtained during annealing: (a, c) at 1 123 K and (b, d) at 1 173 K (b, d). (a) and (b) Inverse pole figure (IPF) maps of \( \gamma \). (c) and (d) Kernel average misorientation (KAM) maps of \( \gamma \).

Fig. 9. Reconstruction of \( \gamma \) from martensite: (a) distribution of variant indices belonging to K–S OR and (b) orientation map reconstructed from Fig. 5(a).

from the tempered martensite structure using the program ROPA ver.9\textsuperscript{31,32} based on the 24 variants of the K–S OR listed in Table 1.

Figure 9 shows the results of the reconstruction of the prior \( \gamma \) grains from the initial tempered martensite IPF map obtained before the reverse transformation. Here, Fig. 9(a) represents the variant distribution under the K–S OR, where the map is colored as a function of the variant number listed in Table 1. It was confirmed that all the variants appeared in the analyzed area. Figure 9(b) shows the prior \( \gamma \) orientation map reconstructed from the initial tempered martensite data shown in Fig. 7(a), in which the boundaries with misorientation angles larger than 15° are displayed by black lines. As shown in Fig. 9(b), the \( \gamma \) grain boundaries and annealing twin can be clearly identified. A comparison of the crystal orientation map of the reversed \( \gamma \) (Fig. 8(a)) with that of the prior \( \gamma \) (Fig. 9(b)) shows good agreement, indicating that most of the reversed \( \gamma \) grains have a nearly K–S OR with the initial martensite laths.

3.5. Mechanism of Austenite Memory and Grain Refinement

In this study, the \( \gamma \) memory phenomenon appeared despite the lack of retained \( \gamma \). The mechanism for the growth and coalescence of the retained \( \gamma \) proposed by Kimmins\textsuperscript{6} and Hara\textsuperscript{7} cannot explain the present results. Moreover, the specimen used in this study contained hardly any cementite because the chromium-carbide (M$_{7}$C$_{3}$) was mainly precipitated as carbide. Hence, the present results also do not agree with Watanabe’s proposal\textsuperscript{18} for a specific OR among
the three phases of ferrite, \( \chi \) and cementite for the occurrence of \( \gamma \) memory. As shown in this study, some reversed \( \gamma \) grains maintained a specific crystallographic relationship through the transformation cycles. It would be acceptable for the \( \gamma \) grains to have formed in accordance with the variant selection mechanism proposed by Nakada \(^{19}\) during the reverse transformation for the selection of a single variant attributed to the relaxation of the local residual stresses in the martensite matrix.

Based on the above results and discussion, a possible mechanism for the \( \gamma \) memory and grain refining after the completion of the reverse transformation is schematically summarized in Fig. 10. As pointed out in previous studies, there are two types of nucleation mechanism of \( \gamma \) from tempered martensite.\(^{6,7,10-12}\) As previously discussed, one is a grain with almost the same crystal orientation as the prior \( \gamma \) and the K–S OR with the initial tempered martensite (type A). This type-A grain corresponds to an acicular grain formed along the lath boundary of the initial tempered martensite (see Fig. 5). The other is a fine \( \gamma \) grain nucleated at the grain boundary or inside the prior \( \gamma \) grain with a different crystal orientation (type B). The type-B grain must be equivalent to the globular \( \gamma \) grain observed in a previous study.\(^6\) It is known that the \( \gamma \) grain nucleates at PAGB and the \( \alpha \)/cementite interface located on a grain boundary, during the reverse transformation from tempered martensite of plain carbon steel.\(^{33}\) However, the present low alloy Ni–Cr–Mo steel mainly precipitated chromium carbide (M\(_7\)C\(_3\)) instead of cementite as carbide. The undissolved chromium carbides were still present when the specimen was reheated in the (\( \alpha + \gamma \)) two phase region (see Fig. 6), after which globular \( \gamma \) grains were hardly formed. This indicates that the \( \alpha \)/chromium carbide interface rarely serves as a nucleation site of globular \( \gamma \). The globular grains seem to preferentially precipitate at PAGB and cementite particles in this steel. Matsuda et al. \(^{11}\) stated that globular \( \gamma \) grains are formed together with the dissolution of cementite particles. Additionally, Hara et al. \(^7\) showed that the amount of globular \( \gamma \) grains increases with the carbon content in steel. Consequently, it is thought that the existence of cementite particles plays an important role in the formation of globular \( \gamma \) grains. In fact, a similar in situ EBSD observation using a ferrite-cementite (bainite) steel revealed that the type-B grains formed predominantly from the initial stages, resulting in a fine polygonal austenite structure.\(^{34}\)

Type-A grains with nearly identical orientations within the prior \( \gamma \) grains grow and coalesce upon impingement during the reverse transformation process, leading to the eventual reconstitution of the original prior \( \gamma \) grain structure. This \( \gamma \) memory phenomenon would appear if the nucleation and growth of the type-A grains are more dominant than those of the type-B grains. The new small type-B grains

Table 1. K–S orientation relationships with 24 variants.

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Fig. 10. Schematic illustration to explain mechanisms of \( \gamma \) memory and grain refinement upon annealing of tempered low alloy steel martensite estimated from present experimental result.
grew even after the completion of the reverse transformation and finally replaced the coarse γ grains reconstituted by the type-A grains. Note that the reconstituted coarse γ grains had a large KAM-value, whereas the type-B grains had a low KAM value (see Fig. 8). The origin of the large KAM value is believed to stem from the martensite lath boundaries with a misorientation of a few degrees. A sub-grain boundary must be formed when the type-A grains with a small misorientation impinge. The type-A grains were invaded by type-B grains, and a fine-grained structure was formed at elevated temperatures. This phenomenon was quite similar to general recrystallization. The point is the competition between the nucleation and growth of type-A and type-B grains, which is dependent on the initial microstructure.

4. Conclusions

The microstructure evolution during reverse transformation at a slow heating rate in a Cr–Ni–Mo steel was investigated. In-situ observations at high temperatures using a SEM/EBSD technique during annealing demonstrated the formation of reversed γ and grain refinement mechanisms. The results obtained can be summarized as follows.

(1) The features of the reversed austenite can be categorized into two types: the reversed austenite grains mainly nucleated along lath boundaries with almost the same crystal orientation as the prior austenite (type A), and the reversed austenite grains nucleated at the prior austenite grain boundaries or inside grains with a different crystal orientation from that of the prior austenite (type B).

(2) The original prior austenite grains before martensitic transformation were reconstituted by the growth and coalescence of the type-A grains. This indicated that the austenite memory phenomenon occurred when the nucleation and growth of the type-A grains were more dominant than those of the type-B grains.

(3) Misorientation existed within the reconstituted austenite grains (impingement and coalescence of type-A grains) at the end of the reverse transformation but little was observed for the type-B grains.

(4) The type-A grains were invaded by type-B grains at higher temperatures (above A_C3) and eventually replaced by a fine grain structure. This phenomenon was similar to recrystallization.

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