Grain Structure of Fe–0.3mass%C–9mass%Ni Steel Processed through $\alpha \rightarrow \gamma \rightarrow \alpha'$ Transformation Caused by Spontaneous Reverse Transformation

Tomoyuki YOKOTA, Masaaki FUJIOKA and Masakazu NIKURA

Steel Research Laboratory, JFE Steel Corp., Kawasaki-ku 1-chome, Mizushima, Kurashiki 712-8511 Japan. 1) Steel Research Laboratory, Nippon Steel Corp., 20-1, Shintomi, Futtsu 293-8511 Japan. 2) Industrial Liaison Office, Okayama University of Science, 1-1, Ridaicho, Okayama 700-0005 Japan.

(Received on October 12, 2004; accepted on January 24, 2005)

$\alpha \rightarrow \gamma \rightarrow \alpha'$ (ferrite $\rightarrow$ austenite $\rightarrow$ martensite) transformation caused by spontaneous reverse transformation (SRT) in Fe–0.3C–9Ni mass% steel has been demonstrated using laboratory rolling mill, and grain structure of the plate has been investigated. SRT is a new process to obtain an ultra refined austenite, and resulting ultra refined transformation products, which occurs due to adiabatic heating caused by deformation at temperatures in ferrite phase field. SRT occurred all through the plate thickness by intensive rolling with 90% reduction and final microstructure was ultra-refined martensite. Mean lineal intercepts of the martensite grains were 0.90 $\mu$m at mid thickness and 0.83 $\mu$m at quarter thickness. Ultra-refined grain structure attributes to enhancement of austenite nucleation due to the intensive rolling before $\alpha \rightarrow \gamma \rightarrow \alpha'$ transformation. The martensite texture after the $\alpha \rightarrow \gamma \rightarrow \alpha'$ transformation caused by SRT composed of RD/$\langle111\rangle$, ND/$\langle111\rangle$, and $\langle111\rangle$ fibers. However, these were not sharp, thus crystallographic grain orientation was quite randomised in spite of the intensive rolling. Two consecutive transformations weakened the initial ferrite rolling texture remarkably. Randomization of the grain orientation was more prominent at the quarter-thickness than at the mid-thickness due to a shear strain.

KEY WORDS: ferrite; austenite; martensite; grain refinement; reverse transformation; adiabatic heating; texture; EBSD.

1. Introduction

There has been a great deal of work on ferrite ($\alpha$) grain refinement as a means towards high performance steels. Deformation of austenite ($\gamma$) prior to $\gamma \rightarrow \alpha + \gamma'$ ($\gamma'$: decomposed $\gamma$) transformation and following accelerated cooling are utilised to enhance nucleation and refine the ferrite grain size. This process has been successfully industrialised as thermo-mechanical control process (TMCP). Recently, many studies have been conducted on ultra fine grained steels to pursue further development of structural steels. Low temperature heavy deformation either during or prior to the transformation, namely ultimate use of the TMCP, is employed to obtain ultra fine ferrite grain size as the most promising way. However, ultimate use of the TMCP generates significant amount of adiabatic heating which causes detrimental effects on ferrite grain refinement, although its effects have been usually neglected so far. In fact, ferrite grain size obtained through the ultimate use of the TMCP was limited by the adiabatic heating. Therefore, rolling pass schedules are controlled to avoid it, or inter-mill water cooling apparatus is introduced to suppress it in case of a tandem mill. The adiabatic heating would reduce chemical driving force for the transformation by raising a transformation temperature. In addition, it would promote recovery of deformed austenite as well as grain growth of newly formed ferrite. Thus, dilemma arises that low temperature deformation is indispensable to obtain ultra fine ferrite grain size, but accompanying adiabatic heating is inevitable.

This paper is about a new process which can overcome this dilemma. The basic idea is an application of the adiabatic heating caused by deformation at ferrite phase field to $\alpha \rightarrow \gamma$ reverse transformation. Beneficial effects of ferrite deformation on austenite nucleation, thus austenite grain refinement can be expected. The new process, so called "Spontaneous Reverse Transformation (SRT)" , has already been demonstrated. Final martensite ($\alpha'$) grain size (prior austenite grain size or packet size) with less than 1 $\mu$m has been obtained through $\alpha \rightarrow \gamma \rightarrow \alpha'$ transformation caused by SRT in Fe–0.3C–9Ni mass% steel. Thus, SRT is the process to have an ultra refined austenite, and resulting ultra refined transformation products such as ferrite, bainite and martensite effectively. It is very important to reveal features of grain structure formed through SRT for the future application of this process to commercial steel products, because it has significant influences on strength, formability and mechanical anisotropy, which are basic properties for structural steels. However, microstructural and crystallographic features of the final transformation...
products processed through $\alpha \rightarrow \gamma \rightarrow \alpha'$ transformation caused by SRT have not been clarified in detail. The aim of this investigation was to demonstrate $\alpha \rightarrow \gamma \rightarrow \alpha'$ transformation caused by SRT in Fe–0.3C–9Ni mass% steel using laboratory rolling mill, and clarify resulting grain structure of the rolled plate.

2. Experimental

Chemical composition of the steel examined is shown in Table 1. Cementite ($\theta$) dissolution temperature and $\mathrm{Ae}_3$ transformation temperature were calculated as 625°C and 665°C, respectively using MTDATA and the SGTE database.\(^{10}\) SRT shown schematically in Fig. 1 was demonstrated using a 1000t laboratory rolling mill. Initial configuration of rolling material was 50 mm thickness $\times$ 50 mm width $\times$ 300 mm length and initial microstructure was martensite with prior austenite grain size of 22 $\mu$m. The material was heated to 550°C which is below the cementite dissolution temperature. It was kept there for 30 min, followed by rolling with a total reduction of 90% under a rolling speed of 50 m/min. The rolling reduction was divided into three passes to lighten the load imposed on the mill and inter-pass time was around 5 s. The plate surface temperature was measured 5 s after the finish-rolling pass to see temperature increase due to adiabatic heating, and it was 667°C beyond $\mathrm{Ae}_3$ transformation temperature. The rolled plate was water quenched immediately after temperature measurement. Plate thickness after the rolling was 7 mm. Specimens for microstructural observation were machined from the rolled plate. Longitudinal sections of the specimens were polished and etched using 3% nital and prepared for the optical microscopy as shown in Fig. 2. Vickers hardness measurement was conducted with 9.8 N along the plate thickness to confirm the occurrence of SRT. The occurrence of SRT can be easily detected by hardness increase, because this steel was designed to have high hardenability and resulting $\gamma \rightarrow \alpha'$ transformation is martensitic. On the other hand, $\alpha \rightarrow \gamma$ transformation due to SRT in this steel occurs in diffusional mechanism.\(^7\)

To reveal macroscopic crystallographic features of the plate, texture analysis has been conducted. Specimens for texture analysis were taken from mid-thickness and quarter-thickness of TD plane of the rolled plate. From these textures were identified as (110)$\langle 110 \rangle$ and (111)$\langle 111 \rangle$ pole figures of austenite and its

Table 1. Chemical composition of the steel examined (mass%).

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Sol.Al</th>
<th>N</th>
<th>Ni</th>
<th>Ti</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.31</td>
<td>0.29</td>
<td>0.86</td>
<td>0.007</td>
<td>0.009</td>
<td>0.033</td>
<td>0.0032</td>
<td>9.33</td>
<td>0.019</td>
<td>0.0014</td>
</tr>
</tbody>
</table>

ODF were obtained as well to clarify austenite grain condition just after SRT (and just before martensitic transformation). In addition, micro texture of the rolled plate has been also investigated using electron backscatter diffraction (EBSD) technique to reveal grain structure of the plate. EBSD images were obtained by FE-SEM (25 kV) equipped with Orientation Imaging Microscopy system. The rolled plate was tempered at 600°C for 30 min to sweep out dense dislocation and obtain clear Kikuchi line pattern and then served for EBSD analysis. EBSD analysis was conducted at mid thickness and quarter thickness of TD plane of the rolled plate as optical micrographs. The conditions in which texture measurement and EBSD measurement were conducted are indicated in Fig. 1.

3. Results and Discussion

3.1. Microstructure and Hardness of The Rolled Plate

Figure 3 shows hardness distribution along the plate thickness. The hardness is over HV600 all through the plate thickness, which means SRT occurs all through the thickness.\(^8\) It is reasonable because observed plate surface temperature which is almost same as the temperature at the mid-thickness\(^8\) was higher than $\mathrm{Ae}_3$ transformation temperature. Figure 4 shows microstructures of the rolled plate taken at mid-thickness and quarter-thickness (un-tempered condition). Both the microstructures show ultra-fine grained martensite formed through $\alpha \rightarrow \gamma \rightarrow \alpha'$ transformation, although grain size and detailed grain structure could not be clarified from these optical micrographs.

3.2. Texture Analysis

ODF at $\phi_y=45^\circ$ section will be shown in following results, because major texture components emerge at this section as shown in Fig. 5. Figure 6 shows ODF of retained austenite at $\phi_y=45^\circ$ section taken from (a) the mid-thickness and (b) the quarter-thickness of the rolled plate. From the positions of intensity maxima, the main components of these textures were identified as \((110)[1\overline{1}1\overline{1}]\) and \((111)[\overline{1}1\overline{1}1]\)
[1 – 12] [0 0 1]α, (1 1 2) [1 – 11]α, and (0 0 1) [0 – 10]α, at the both locations. Maximum orientation density f(α) value in whole Euler space were 3.9 at the mid-thickness and 1.9 at the quarter-thickness. Figure 7 shows ODF of martensite at φ2 = 45° section taken from (a) the mid-thickness and (b) the quarter-thickness of the rolled plate. From the positions of intensity maxima, the main components of the texture at the mid-thickness were identified as (1 1 1) [1 1 0]α, (3 3 2) [1 – 1 0]α, (1 1 5) [1 – 1 0]α, and (0 0 1) [1 1 0]α. Thus, RD//(1 1 0)α and ND//(1 1 1)α fibers are developed. RD//(1 1 0)α and ND//(1 1 1)α fibers are also developed at the quarter thickness as at the mid-thickness. However, they are very weak and orientation distribution is rather randomized at the quarter-thickness in accordance with the austenite texture in Fig. 6. Maximum orientation density f(α) value in whole Euler space were 3.0 at the mid-thickness and 1.8 at the quarter-thickness.

Observed maximum f(α) values are quite small in comparison to those reported in heavily rolled ferrite texture or ferrite texture transformed form austenite heavily rolled at low temperature, which is typically ranging from 5 to 10 or more. Thus, sharp texture was not developed after SRT even in the mid-thickness, in spite of the intensive rolling with 90% reduction. This point is a characteristic feature of the martensite texture formed through α→γ→α′ transformation caused by SRT. G. Bruckner et al. investigated transformation textures during diffusional α→γ→α transformation in low alloyed steel and reported that α→γ transformation degraded an initial texture sharpness remarkably and following γ→α transformation also weakened it, although main feature of the original texture was conserved through the transformations. It is reasonable to suggest that initial texture is degraded by two consecutive transformations: α→γ→α′ transformation, because of high (24 hold) multiplicity of transformation variants.

Origin of the observed austenite texture and final martensite texture was discussed as follows. Because rolling started at ferrite phase field and then reverse transformation take place due to adiabatic heating, the texture must be originally developed during ferrite rolling before SRT, and then austenite and martensite transformed from austenite must inherit the texture one after another. There is no direct evidence of initial ferrite texture formed before SRT. It is well-known fact that RD//(1 1 0)α and ND//(1 1 1)α fibers are developed during cold or warm rolling of ferrite. Then, it is reasonable to suppose that initial ferrite texture are composed of both of the fibers. The initial texture will be inherited to a transformation product with a specific lattice correspondence, namely Kurdjumov–Sachs (K–S) relationship as follows.

\[ \{1 1 1\} // \{1 1 0\} α, \langle 0 1 1 \rangle, // \{1 1 1\} α \]

Figure 8 shows intensity maxims of austenite texture, which are transformed from several ferrite texture components, lie in the two fibers using K–S lattice correspondence assuming all transformation variants are activated. Experimental austenite texture can be reasonably explained by calculated intensity maxims. Thus, observed austenite texture was revealed to be inherited from initial ferrite rolling texture through the α→γ reverse transformation (SRT).

It should be noted that austenite could be rolled to some extent during or after SRT and form austenite rolling texture superimposing over the texture inherited from original ferrite rolling texture. It is not simple to distinguish austenite rolling texture from the austenite texture inherited from ferrite rolling texture, because the components of the both textures are in common. Actually, recrystallised austenite texture component: (0 0 1) [0 – 1 0]α and non-recrystallised austenite texture components: (1 1 2) [1 – 1 1]α, and (1 1 0) [1 – 1 2]α are all lie in the austenite texture inherited from ferrite rolling texture. It is possible to guess how much austenite was deformed by knowing development behaviour of Cu type rolling texture with rolling reduction. It is clear that {1 1 0} (1 1 2) component develops sharply as rolling reduction increases. If austenite was rolled heavily after SRT, (1 1 0) [1 – 1 2] component must be developed sharply. However, experimental (1 1 0) [1 – 1 2] component is not remarkable in comparison to other texture components. In fact, orientation density f(α) of the component was 1.9 at
The mid-thickness and 1.8 at the quarter-thickness. The other point is, it is not likely for austenite grains to recrystallise and form recrystallisation texture in such a low temperature region. Then, it can be concluded that rolling was mainly conducted in ferrite phase field and the origin of the austenite texture is inheritance of the initial ferrite rolling texture. It is worth noting that a pole figure which implies deformation in the austenite phase field was observed at a mid-thickness in another Fe–0.3C–9Ni mass% steel plate processed through SRT, in which rolling conditions were almost same as this study, although ODF had not been re-

Fig. 4. Microstructures of the rolled plate taken at (a) the quarter-thickness and (b) the mid-thickness.

Fig. 5. Major texture components in $\phi_1=45^\circ$ section of Euler space in Bunge's notation.

Fig. 6. ODF of retained austenite at $\phi_2=45^\circ$ section taken from (a) the mid-thickness and (b) the quarter-thickness of the rolled plate.

Fig. 7. ODF of martensite at $\phi_2=45^\circ$ section taken from (a) the mid-thickness and (b) the quarter-thickness of the rolled plate.

Fig. 8. Intensity maxims of austenite texture components transformed from several ferrite texture components lie in ND//111 and RD//110 fibers through the K–S lattice correspondence.

Fig. 9. Intensity maxims of final martensite texture components transformed from several austenite texture components through the K–S lattice correspondence.
ported. Thus, exact amount of deformation in the austenite phase field remains unclear.

Figure 9 shows intensity maxims of final martensite texture, which are transformed from experimentally obtained austenite texture components using K-S lattice correspondence in the same manner as before. Experimentally obtained martensite texture can be well explained again by calculated intensity maxims. Thus, observed martensite texture is inherited from austenite texture through the \( \gamma \rightarrow \alpha' \) transformation after SRT. It is evident that a set of the most prominent martensite textures \((111)\langle110\rangle_\alpha\) and \((115)\langle1\overline{1}0\rangle_\alpha\) can be come from \((110)\langle1\overline{1}1\rangle_\gamma\), \((110)\langle001\rangle_\gamma\), and \((001)\langle0\overline{1}0\rangle_\gamma\), which are also the most prominent austenite texture components. Thus, the final martensite texture after SRT can be rationally explained as transformation texture inherited from initial ferrite rolling texture through \(\alpha \rightarrow \gamma \rightarrow \alpha'\) transformation.

3.3. EBSD Analysis

Figure 10 shows EBSD images and corresponding inverse pole figures taken from the rolled and tempered plate. The colours of the images indicate ferrite grain orientation parallel to ND direction. Black coloured region indicates precipitated cementite and austenite on martensite grain boundaries by the tempering at 600°C. Majority of the ferrite grains are ND/\((111)\langle001\rangle_\alpha\) showing from blue colour to red colour at the mid thickness and this is consis-

![Fig. 10. EBSD images taken from (a) mid-thickness and (b) quarter-thickness.](image)

![Fig. 12. EBSD image taken from quarter-thickness showing ND/\((110)\alpha\) colony.](image)
the quarter-thickness and therefore crystal orientation at the quarter thickness is almost at random. This result corresponds to very weak texture observed at the quarter-thickness. Thus, the results of EBSD analysis and texture analysis are compatible.

It is well known that ND//\{110\}_{a} rolling texture develops with shear strain due to roll friction.\(^{17-20}\) Sakai et al.\(^{19}\) studied inhomogeneous texture formation along a plate thickness in hot rolled ferritic steels and clearly showed ND//\{110\}_{a} texture developed in accordance with an amount of shear strain. It can be seen from their paper that because ND//\{110\}_{a} texture develops close to plate surface and conventional ND//\{111\}_{a}−\{001\}_{a} rolling texture develops at mid-thickness, very weak texture region appears between those two regions. Very randomised grain structure including ND//\{110\}_{a} grains at the quarter-thickness obtained in this study can be qualitatively explained by this phenomenon, because equivalent strain remarkably increases toward the plate surface due to shear strain component introduced by the rolling pass conducted in this study.\(^{21}\) It can be understood that the random grain structure at the quarter-thickness would result from initial ferrite rolling texture originally developed under the existence of shear strain before \(\alpha \rightarrow \gamma \rightarrow \alpha'\) transformation.

Majority of the ferrite grains show equiaxed morphology. Mean linear intercept of the ferrite were measured as 0.73 \(\mu m\) at the mid thickness and 0.49 \(\mu m\) at the quarter thickness from the EBSD images. These values were measured without accounting precipitated austenite grain region (black coloured region), and thus for ferrite grain size after 600°C tempering. Actual martensite grain size before tempering should be much larger, because the austenite grain region should be originally occupied by fresh martensite grain. To evaluate martensite grain size before tempering properly, austenite grain region is assumed to be one of the surrounding ferrite grain’s orientations. Thus, mean linear intercept of martensite grain size before tempering was estimated as 0.90 \(\mu m\) at the mid-thickness and 0.83 \(\mu m\) at the quarter-thickness. It is of importance to discuss what “martensite grain” means, because conventional stratum of martensitic structure such as lath, block and packet could not be observed in this study. It might be prior austenite grain size or packet size. Packets are group of laths with the same habit plane.\(^{21}\) In general, martensite packet size is in proportion to prior austenite grain size and its ratio (prior grain size/packet size) ranges from 2 to 3 in carbon steels.\(^{22}\) The ratio approaches unity as prior austenite grain size decreases.\(^{21}\) Thus, martensite grain size can be regarded as almost prior austenite grain size. This is the reason why the stratum of martensitic structure could not be observed in this study.

Possible microstructural change through \(\alpha \rightarrow \gamma \rightarrow \alpha'\) transformation caused by SRT is schematically illustrated in Fig. 11, taking into account the results of EBSD analysis. There is no direct evidence of grain structure formed during ferrite rolling before SRT. However, it can be inferred from those obtained through similar rolling conditions, in which SRT does not take place. Ohmori et al.\(^{23}\) investigated ferrite microtexture formed through intensive rolling of martensitic or bainitic steel at similar temperature range \((\alpha + \theta \text{ region})\) in which SRT does not take place. RD//\{110\}_{a} and ND//\{111\}_{a} fibers were developed as we assumed before, and blue and red colored ND//\{111\}_{a}−\{001\}_{a} grains elongated layer by layer along rolling direction in which subgrains forms are prominent in EBSD images.\(^{23}\) The grain structure formed during ferrite rolling before SRT in this study should be similar to it. At the quarter-thickness, ND//\{110\}_{a} grains should be existed due to the shear strain as described before. Presumably, these ND//\{110\}_{a} grains would be also elongated as ND//\{111\}_{a}−\{001\}_{a} grains, because green colored fine ND//\{110\}_{a} grains seems to aggregate within prior elongated grain after \(\alpha \rightarrow \gamma \rightarrow \alpha'\) transformation as shown in Fig. 12. Thus, grain structure formed during ferrite rolling before SRT likely to be layered microstructure at this stage. SRT should start from this deformed microstructure. Austenite grains nucleate at precipitated cementite on sub grain boundary of the deformed matrix and grow in diffusion mechanism.\(^{21}\) Intensive rolling at ferrite phase region has an important effect on ferrite subgrain refinement, thus austenite nucleation.\(^{6}\) The layered microstructure would be extinguished in this \(\alpha \rightarrow \gamma\) transformation and ultra-fine equiaxed austenite grain structure would be formed at this stage. Main texture component of newly formed austenite is ND//\{110\}_{a} transformed from ND//\{111\}_{a} through K-S lattice correspondence as shown in Fig. 8. Then finally, \(\gamma \rightarrow\)
\(\alpha'\) martensitic transformation take place, in which each martensite grain forms from one prior austenite grain. Main texture component of final martensite is back to ND/\((111)_{\alpha}\) again transformed from ND/(110) \(_{\alpha}\) through K–S lattice correspondence as shown in Fig. 9, although orientation distribution is randomised through these two consecutive transformations.

4. Summary

\(\alpha \rightarrow \gamma \rightarrow \alpha'\) transformation caused by SRT in Fe–0.3C–9Ni mass% steel has been demonstrated using laboratory rolling mill, and grain structure of the plate has been investigated. Obtained results are summarised as follows.

(1) SRT occurred all through the plate thickness and final microstructure was ultra-refined martensite. Majority of the martensite grains showed equiaxed morphology and final microstructure was ultra-refined martensite. Majority of the martensite grains showed equiaxed morphology and mean lineal intercepts of the grains were 0.90 \(\mu\)m at the mid thickness and 0.83 \(\mu\)m at the quarter thickness. Ultra-refined grain structure attributes to enhancement of austenite nucleation due to intensive rolling before \(\alpha \rightarrow \gamma\) transformation.

(2) The martensite texture after \(\alpha \rightarrow \gamma \rightarrow \alpha'\) transformation caused by SRT composed of RD/(111) \(_{\alpha}\) and ND/(110) \(_{\alpha}\) fibers. However, these were not sharp, thus crystallographic grain orientation was quite randomised in spite of the intensive rolling with 90% reduction. Two consecutive transformations weakened the initial ferrite rolling texture remarkably. Randomization of the grain orientation was more prominent at the quarter-thickness than at the mid-thickness due to a shear strain.

(3) The martensite texture after \(\alpha \rightarrow \gamma \rightarrow \alpha'\) transformation caused by SRT can be rationally explained as transformation texture inherited from initial ferrite rolling texture through \(\alpha \rightarrow \gamma \rightarrow \alpha'\) transformation.

Acknowledgement

The experimental work had been carried out from 1997 to 2001 fiscal year as a part of research activities of Ferrous Super Metal project. Financial support from NEDO (New Energy and Industrial Technology Development Organization) is gratefully acknowledged.

REFERENCES