Grain Refinement of C–Mn Steel to 1 μm by Rapid Cooling and Short Interval Multi-pass Hot Rolling in Stable Austenite Region

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A noble grain refinement method to attain ferrite grain size of 1 μm in plain C–Mn steel by multi-pass hot rolling in a stable austenite temperature region has been explored. 0.1%C–1%Mn and 0.16%C–0.7%Mn steels were finish-rolled at about A_{e3} and then rapidly cooled. Special emphases have been placed on the interval, Δt , between the finish of rolling and the start of water spray cooling of more than $1000^\circ\text{C} \cdot \text{s}^{-1}$ in cooling rate and the interval between the last two rolling passes. By reducing Δt from 0.5 to 0.05 s alone, the grain sizes near the sheet surface and in the central region were markedly reduced to 1.3 and 2.2 μm respectively. By reducing Δt and the rolling pass interval together, a further reduction in grain size was then achieved. Consequently, the steel sheet in which the ultrafine grain structure of about 1 μm in grain size penetrated from the sheet surface to the depth of a quarter of the sheet thickness and the grain size in the central region was well below 2 μm was obtained. The transformation to the ultrafine ferrite is considered to be static and from deformed γ . The detailed grain refinement mechanisms are discussed based on the microstructure observation of quenched C–Mn steels and a fcc Ni–30%Fe alloy as well as the calculation of prior γ texture by the misorientation distribution function method.

KEY WORDS: grain refinement; ferrite transformation; C–Mn steel; multi-pass rolling; ultra-fast cooling; texture.

1. Introduction

Reduction in ferrite (α) grain size increases the strength of steel linearly to the inverse of the square root of grain size, as has been pointed out by Hall and Petch.^{1,2)} At the same time, other properties such as toughness and ductility are improved without microalloying elements. An effective method known to obtain such benefits owing to the grain refinement is the thermomechanical controlled process (TMCP)³⁾ that has been widely applied in modern commercial hot rolling mills. However, it is commonly agreed that TMCP possesses the lower limit of grain size around 3 to 5 μm for plain C–Mn steel⁴⁾ regardless of the amount of strain imparted into austenite (γ) by rolling. The Hall–Petch relation predicts that a further grain size reduction, for example, to 1 μm may lead to as much increase in strength as 350 MPa. Therein exists a strong motivation for researchers to investigate the methodology to further reduce grain size. A variety of methods are therefore researched throughout the world to synthesize a new generation of fine-grained high strength steel.⁴⁾

The recent investigation on grain refinement of steel has been mainly two-fold. One is on the basis of the so-called continuous recrystallization of α .⁵⁾ As materials undergo severe deformation over 2 to 4 in strain at a warm α tem-

perature, the accumulated strain can create new high angle boundaries due to crystal rotation and thermally activated migration of dislocations. Then new α grains may appear without nucleation. Based on this phenomenon, α grain size below 1 μm has been reported for many kinds of steel using various processes, such as mechanical milling^{6,7)} accumulative roll-bonding,⁸⁾ equal channel angular pressing,⁹⁾ high pressure torsion¹⁰⁾ and heavy plate rolling.¹¹⁾

The other route of investigation has been based on the phenomenon called the dynamic strain-induced transformation (DSIT).^{12–15)} In early 1980's, Priestner¹²⁾ first observed a small amount of extremely fine α grains that precipitated along γ grain boundaries during hot rolling at a relatively low temperature. Yada and his co-workers¹³⁾ later reported that the microstructure consisting of even finer α grains of 1 to 2 μm appeared in plain C–Mn steel as the amount of strain exceeded unity in compression deformation tests at temperatures mostly below A_{e3} . Hodgson and his co-workers^{14,15)} also reported similar fine structures that appeared in layers near sheet surfaces as largely undercooled γ was deformed by single-pass hot rolling. These phenomena are now commonly recognized as DSIT, while different researchers use slightly different nomenclature. In DSIT, a large strain imparted in undercooled γ causes a dramatic increase in the number of nucleation sites for α , so that a

dynamic transformation (*i.e.* transformation during deformation) takes place resulting in a remarkable grain refinement. Although those methods are of considerable scientific importance, there are still difficulties for them to be commercially applied due to a large deformation resistance at required low temperatures and complexity of processes.

The question that arises in the course of the above investigations is whether or not the lower bound of α grain size for TMCP is inevitable as hot rolling is carried out above A_{e3} . The ultrafine α formation by DSIT implies that, as a sufficient amount of strain is kept remained in γ , the subsequent transformation may lead to a dramatic reduction in grain size regardless of rolling temperature. The aim of our research, therefore, has been to investigate a method that reduces the lower bound of α grain size in TMCP with rolling temperature being kept above A_{e3} . The results obtained have been only partly published in some proceedings of international conferences.^{16–24)} Thus, in this article are given a thorough description of the explored grain refinement method and therein operating mechanisms. The investigated method is composed of short interval multi-pass hot rolling and subsequent ultra-fast cooling. Firstly, the effects of the ultra-fast cooling on grain refinement are described, which are the leading knowledge to the present grain refinement. Then the combined effects of the ultra-fast cooling and the reduction in rolling pass interval are presented, of which process has been developed in a Japanese national project, “Production Technologies for Environment-conscious Ultrafine-grained Steel”,²⁵⁾ based on the above earlier knowledge and has been called the Super-Short interval Multi-pass Rolling (SSMR).^{18,26)} Finally, the grain refinement mechanism is discussed based on the experimental results as well as texture transformation calculation by the misorientation distribution function method (MODF).^{27,28)}

2. Experimental Procedure

The hot rolling simulator used in this experiment is schematically represented in Fig. 1. It consists of three rolling stands designated as F1 to F3 and water-cooling devices. The cooling device after F3 was designed to attain a large cooling rate over $1000^{\circ}\text{C}\cdot\text{s}^{-1}$ for the samples under investigation and to control the intervening time period Δt between the last rolling pass and the beginning of the rapid cooling. The Δt in this experiment is specifically defined as

the period between the instance of the exit from the last rolling and the instance at which high-pressure water jets first make contact with samples. The cooling devices between the rolling stands were used to suppress adiabatic deformation heating when it was necessary to maintain sample temperature.

The steels with chemical compositions listed in Table 1 were hot forged and machined into the plates of 30 or 35 mm in thickness. They were reheated to 1000°C and rough-rolled with 3 rolling passes using the F1 stand. Then they were successively finish-rolled through all the stands with the rolling pass intervals ranging from 0.17 to 1 s. The reduction of each pass was 30 to 50%, where the total reduction was 95 to 97% resulting in sheets of 1.0 to 1.3 mm in thickness. Finishing temperature was controlled to be around the A_{e3} of the samples; the paraequilibrium A_{e3} calculated for both the steels A and B by THERMOCALC was 818°C . The finish-rolled samples were then rapidly cooled to temperatures around 650°C , at which the transformation from γ to α was known to become active, or to the room temperature to investigate the structure of γ prior to the transformation. The cooling rate was over $1000^{\circ}\text{C}\cdot\text{s}^{-1}$ as aforementioned, and the Δt was varied in the range from 0.05 to 0.5 s. The steel A once rapidly cooled to about 650°C was then air-cooled for several seconds being followed by the cooling to the room temperature at the rate of about $200^{\circ}\text{C}\cdot\text{s}^{-1}$, while the steel B cooled to about 620°C was then air-cooled to the room temperature.

The microstructural evolution in deformed γ during hot rolling was simulated using an fcc Ni–30%Fe alloy with a chemical composition listed in Table 1. This alloy has a stacking fault energy (S.F.E.) similar to that of γ iron and low carbon steel,²⁹⁾ and this alloy is known to behave similarly to low carbon steel during hot working.³⁰⁾ The rolling and cooling conditions for the Ni–Fe alloy were the same as those described above except for finishing temperature. Two-pass compression tests using Thermecmaster were carried out to determine the softening rates of the Ni–Fe alloy and the steel B, and the temperature of 900°C at which the softening rate of the Ni–Fe alloy was equivalent to that of the steel B at 800°C was chosen for the finishing temperature. As for the compression tests, samples were first heated to 1000°C , and then they were twice compressed with the reductions of 60 and 20% after cooling to the desired temperatures. The softening rates were determined as the ratio

Fig. 1. Schematic representation of experimental hot-rolling simulator.

Table 1. Chemical compositions of specimens (mass%).

	C	Si	Mn	P	S	Al	N	Ni
Steel A	0.10	0.05	1.11	0.018	0.003	0.017	0.0035	-
Steel B	0.15	0.01	0.75	0.020	0.002	0.022	0.0022	-
Ni-30%Fe alloy	0.002	-	-	0.002	<0.001	-	0.002	70.3

of the difference between the maximum stress at the first compression, σ_m , and the yield stress of the second compression to the difference between σ_m and the yield stress of the first compression.

The microstructures and crystallographic textures of the samples were examined via transmission electron microscopy (TEM), scanning electron microscopy (SEM), electron back scattering diffraction analysis (EBSD) and X-ray diffraction techniques (XRD). For the texture analysis, orientation distribution functions (ODF) were calculated using three incomplete pole figures determined by XRD, (110), (200) and (211) pole figures for bcc and (111), (200) and (220) pole figures for fcc. These measurements were made 0.05 to 0.1 mm below the sample surface, a quarter of sheet thickness below the surface and in the central region.

3. Misorientation Distribution Function Method (MODF)

Since much information on microstructure evolution in γ during deformation is present in the crystallographic texture of γ , the texture of prior γ has been analyzed by MODF.^{27,28)}

MODF is a calculation method developed based on the representation of textures of materials by the orientation distribution function (ODF), and it relates the series expansion coefficients of the ODF of a parent phase, ${}^{\gamma}C_{\lambda}^{\mu\nu}$, to those of its product phase, ${}^{\alpha}C_{\lambda}^{\mu\nu}$, with a set of linear equations shown below,²⁸⁾

$$\begin{aligned} {}^{\alpha}C_{\lambda}^{\mu\nu} = & \sum_{\lambda_1=0}^{\infty} \sum_{\mu=1}^{M(\lambda_1)} \sum_{\nu_1=1}^{N(\lambda_1)} {}^{\gamma}C_{\lambda_1}^{\mu\nu_1} \\ & \times \left[\sum_{\lambda_2=0}^{\infty} \sum_{\nu_2=1}^{N(\lambda_2)} \sum_{m=-\lambda}^{\lambda} \sum_{s=-\lambda}^{\lambda} \rho_{\lambda_2}^{r\nu_2} A_{\lambda_1}^{m\mu} (\lambda_1 \lambda_2 m r | \lambda s) \right. \\ & \left. \times \{ \lambda_1 \lambda_2 \nu_1 \nu_2 | \lambda \nu \} T_{\lambda}^{\mu s*}(\Delta g) \right] \dots\dots\dots(1) \end{aligned}$$

$$s = m + r, s \leq \lambda, |\lambda_2 - \lambda_1| \leq \lambda \leq |\lambda_1 + \lambda_2|$$

where $T_{\lambda}^{\mu s}(g)$'s and $A_{\lambda_1}^{m\mu}$'s are the symmetrically invariant functions and related coefficients defined for the sample with orthorhombic symmetry (*i.e.* rolling symmetry), and $(\lambda_1 \lambda_2 m r | \lambda s)$ and $\{ \lambda_1 \lambda_2 \nu_1 \nu_2 | \lambda \nu \}$ are Clebsch–Gordan coefficients and related coefficients defined by Bunge *et al.* respectively.²⁸⁾ Δg is the crystal rotation by the phase transformation and $\rho_{\lambda_2}^{r\nu_2}$'s are the expansion coefficients of a variant selection function $\rho(g)$ by $T_{\lambda}^{\mu s}(g)$'s. The Eq. (1) are solvable for ${}^{\gamma}C_{\lambda}^{\mu\nu}$, if ${}^{\alpha}C_{\lambda}^{\mu\nu}$, Δg and $\rho(g)$ are known. Therefore, if the series expansion coefficients of textures of α are determined from XRD data, the texture of the prior γ before transformation can be deduced.

In the present calculation are assumed the Kurdjumov–Sachs (KS) orientation relationship³¹⁾ between γ and α and a variant selection rule, in which α particles nucleated on high angle boundaries in γ hold the KS or near-KS relationship with two neighboring γ grains at the same time as shown in Fig. 2. Let us call this variant selection rule the double KS relation. The $\rho(g)$ for this selection rule may be

Fig. 2. Schematic representation of the double KS relation.

expressed as shown below,

$$\begin{aligned} \rho(g) = & \frac{\omega}{21} \sum_B f_{\gamma}(\Delta g^{-1} \cdot g_B^c \cdot \Delta g \cdot g) \\ & - \frac{\omega}{21 \cdot 24} \sum_{A,B} f_{\gamma}(\Delta g^{-1} \cdot g_B^c \cdot \Delta g \cdot g_A^c \cdot g) \end{aligned} \dots\dots(2)$$

where g^c represents the 24 rotation matrices of the cubic symmetry group, and f_{γ} and g represent the ODF of γ and the orientation of γ respectively. The $\rho(g)$ is a weight function by which the probability of occurrence of variants is determined, and 24 different transformation variants are described by 24 equivalent parent orientations that coexist in the entire orientation space because of the cubic symmetry in γ crystals, $g^c \cdot g$; *i.e.*, in general $\rho(g) \neq \rho(g^c \cdot g)$ when $g^c \neq 1$. Δg does not change for different variants in the calculation.

The $\Delta g^{-1} \cdot g_B^c \cdot \Delta g \cdot g$ in the first term on the right hand side of the Eq. (2) expresses the orientation of γ , with which an α variant transformed from γ having the orientation of g can hold the KS relationship at the same time (see Fig. 2). Since $f_{\gamma}(\Delta g^{-1} \cdot g_B^c \cdot \Delta g \cdot g)$ is the probability to find such γ gains, the first term represents the probability by which the α variant can precipitate under the double KS relation. Note that the summation for B is taken only for 21 g^c 's, whereas that for A is taken for all of the 24 g^c 's. This is because, if g_B^c is, for instance, unity, $\Delta g^{-1} \cdot g_B^c \cdot \Delta g \cdot g$ is equal to g . It then corresponds to the case where the boundary on which α precipitates disappears. Likewise, two other g_B^c 's correspond to the case where α precipitates on low angle boundaries (about 10.5° of misorientation). Hence, these three cases are omitted in the summation; it is suggested that α mostly precipitates on high angle grain boundaries as will be described later in the Sec. 6. The last term in the Eq. (2) is to satisfy the material conservation law shown below,

$$\frac{1}{24} \sum_A \rho(g_A^c \cdot g) = 1$$

The $\rho(g)$ in the Eq. (2) is well-defined (in a physical sense) only in the range of ω from 0 to around 1, and the parameter ω can be regarded as the approximate volume fraction of α that precipitates under the double KS relation. The ω in the present calculation is 0.7, by which the texture of α in a hot-rolled steel sheet has been extremely well reproduced from the texture of retained γ in the same sample.³²⁾ The details of the above variant selection model and the calculation method will be reported elsewhere.³²⁾

4. Experimental Results

4.1. Grain Refinement by Ultra-fast Cooling

The steel A was hot rolled and rapidly cooled to temperatures around 650°C with a variety of Δt to understand how the intervening period between hot rolling and cooling influenced grain refinement. The rolling pass interval was relatively large from 0.6 to 1 s, and the reductions for the last three passes were 40, 40 and 50% in temporal order.

Figures 3 and 4 show the dependence of α grain size on Δt and the corresponding SEM micrographs of the steel A respectively. When Δt is 0.5 s, the average grain diameter is about 3 μm almost irrespective of the depth from sheet surfaces, although the grain size near sheet surfaces (0.1 mm below the sample surface) is slightly smaller than that in the central region. This grain size is very close to the lower limit of grain size reported for C–Mn steel by conventional TMCP. Therefore, with the large Δt of 0.5 s under which TMCP is conventionally performed, the grain size does not reduce to below its known limit, no matter how large hot deformation and how fast subsequent cooling are applied within the conditions in this study.

However, by decreasing Δt , a remarkable grain refinement occurs. When Δt reduces to 0.05 s, the grain sizes near the sheet surface and in the central region decrease to about 1.3 and 2.2 μm respectively, as shown in Figs. 3 and 4. Whereas this reduction in grain size is gradual in the central region, it is rather abrupt with a large drop between 0.05 and 0.2 s in Δt near the sheet surface. The obtained α grains are mostly equiaxed, and there is the tendency that

Fig. 3. Change in α grain size as a function of Δt for the steel A.

Fig. 4. SEM cross-sectional micrographs for the steel A hot-rolled and rapidly cooled to about 650°C.

the finer the microstructure, the more equiaxed it becomes. The TEM micrograph shown in Fig. 5 reveals the details of the equiaxed fine grain structure. Many of the grains in the surface region are as small as 1 μm , and elongated grains scarcely exist. It is also revealed in this figure that little dislocation is present in the α grains, which evidences that the grains have not undergone deformation once formed. On the boundaries between such extremely refined grains, cementite particles of which diameter is about 0.1 μm exist.

4.2. Combined Effects of Rapid Cooling and Short Interval Multi-pass Rolling (SSMR)

A further grain refinement by incorporating short interval multi-pass rolling with the rapid cooling was then investigated using the steel B. The rolling pass interval between F2 and F3 was reduced down to 0.17 s with Δt being equal to 0.05 s. The reductions of the last three passes in this case were 50, 40 and 50% in temporal order.

The results are shown in Fig. 6. As the rolling pass interval is the same as that in the previous section (*i.e.* from 0.5 to 1 s), the grain size of the steel B is nearly equal to that observed for the steel A with reduced Δt . The obtained grain size for the steel B in this condition is from 1.3 to 2.2 μm depending upon the depth from sheet surfaces, which confirms the grain refinement effect of the ultra-fast cooling described in the previous section (see Fig. 3). As the rolling pass interval is reduced from over 0.5 s down to 0.17 s, a further grain refinement takes place. This grain refinement almost equally occurs in any part of cross-section

Fig. 5. TEM micrograph of the steel A hot-rolled and rapidly cooled to about 650°C at Δt of 0.05 s.

tions, and the average grain diameter is thus reduced to 0.87, 1.1 and 1.4 μm near the sheet surface, a quarter of the thickness below the surface and in the central region, respectively. Hence the steel sheets, in which the ultrafine grain structure of about 1 μm in grain size penetrates to the depth of a quarter of the total thickness and the grain size in the central region is well below 2 μm , can be obtained by the present grain refinement method.

The grain structure and grain boundary angle distributions typical of such an extremely grain-refined steel B are shown in Figs. 7 and 8. The grains near the sheet surface are mostly below 1 μm in diameter and remarkably equiaxed. In this area, about 90% of grain boundaries have misorientation angle of greater than 15°, *i.e.* of high angle. In the central region, the presence probability of high angle grain boundaries is about 80%, and grains are slightly elongated in the rolling direction.

4.3. Analyses on Grain Refinement Mechanism

4.3.1. Rapidly Quenched Microstructure

In Fig. 9 are shown the SEM micrographs of the steel A prepared in the same way as those for the samples shown in Fig. 4 but being rapidly cooled to the room temperature to bypass ferrite transformation during cooling. Martensitic structures with a small amount of α on prior γ grain boundaries are observed except for the microstructure shown in Fig. 9(a). The presence of a great amount of martensite evidences that the structure before cooling is predominantly of γ . Thus, it clearly indicates that the fine α grains of 2 to 3 μm in diameter, observed in Figs. 4(b) to 4(f), are mainly

due to the transformation to α after the cooling to 650°C, *i.e.*, due to a static transformation. Furthermore, from the distribution of the small α grains on prior γ grain boundaries, it is recognized that the prior γ grains in the central region are largely elongated in the rolling direction (Figs. 9(d) to 9(f)), whereas those near the sheet surface are more or less spherical (Figs. 9(b) and 9(c)). Hence, the static transformation that occurs in the central region is most likely the one from deformed γ , while that occurring near the sheet surface at Δt over 0.3 s is the one from recrystallized γ .

In contrast, the microstructure shown in Fig. 9(a) is exceptionally consisted of fine α grains of about 1 μm diameter despite the rapid quenching to the room temperature. Two opposite explanations are therefore drawn for the formation mechanism of the ultrafine α near the sheet surface shown in Fig. 4(a). One explanation is simply that the ultrafine grains of about 1 μm size are formed before the rapid cooling, *i.e.*, due to dynamic transformation during hot deformation (or nearly dynamic one including transformation

Fig. 6. Change in α grain size for the steel B as a function of time interval between last two rolling passes.

Fig. 7. Grain structure of the steel B rapidly cooled to about 620°C at Δt of 0.05 s after hot-rolling with short pass interval of 0.17 s. (a) Surface region and (b) central region.

Fig. 8. Distribution of misorientation angle between individual α grains. (a) Surface region and (b) central region.

Fig. 9. SEM micrographs of the steel A rapidly cooled to the room temperature. Broken lines fringe prior γ grains.

Fig. 10. TEM micrographs of Ni-30%Fe alloy hot-rolled with short rolling pass interval of 0.17 s and rapidly cooled to the room temperature at Δt of 0.05 s (a) near the sheet surface and (b) in the central region.

during the short period of 0.05 s after hot rolling). The other explanation is that the static transformation to the ultrafine α is so fast that it cannot be bypassed by the rapid quenching. We will return to this point later in the Sec. 6.

4.3.2. Ni-30%Fe Model Alloy

Another critical aspect to be analyzed on the transformation to the ultra-fine α is the form of nucleation sites in γ . In this respect, hot rolled Ni-30%Fe model alloys were examined in microstructures and textures. Finishing temperature of hot rolling was chosen to be 900°C at which the model alloy showed equivalent softening rates to the steel B. In Fig. 10 are shown TEM micrographs of a surface region of the model alloy that has been hot-rolled with a short rolling pass interval between F2 and F3, 0.17 s, being followed by the rapid cooling of 0.05 s in Δt , which are the close conditions to those for the sample shown in Fig. 7 except for finishing temperature. The planes of observation are normal to the transverse direction. Almost homogeneous distribution of equiaxed and cellular arrays of dislocations are observed, which are very similar to those observed in cold-rolled fcc alloys³³⁾ as well as hot-rolled Ni-30%Fe.^{34,35)} The average cell diameter is approximately 0.4 μm . Although the misorientation angle between neighboring cells is mostly very small, some of the cell walls in the present experiment show large misorientation over 10° as shown in Fig. 10(b). Such high angle cell walls appear to be rather sharp walls in the TEM micrographs. It is noteworthy that a dense distribution of dislocations has been observed inside all the cells. Whereas some cells in Fig. 10 seemingly include few dislocations, the tilting of TEM foil planes has revealed an abundant presence of dislocations within the cells. Therefore, indicator of obvious recrystallization has been seldom present.

Figure 11 shows the EBSD analyses on the section normal to the transverse direction for the same sample as that in Fig. 10. While the above homogeneous distribution of cell structures cannot be clearly seen probably because the change in orientation across most of cell boundaries is too gradual to be detected as boundaries in the EBSD analysis, elongated grains in the rolling direction, small equiaxed grains as well as inter-granular low angle boundaries are observed. The small equiaxed grains are likely to correspond to the high angle cell walls seen in the TEM photographs in Fig. 10. Some of the low angle boundaries are aligned in the directions about 30° from the rolling direction like microbands, and ledges on the high angle grain

Fig. 11. Boundary maps for the Ni-30%Fe alloy shown in Fig. 10 by EBSD (a) near the sheet surface and (b) in the central region.

boundaries are also seen. The grains near the sheet surface are finer and less elongated in the rolling direction than those in the central region, and the volume fraction of equiaxed grains near the sheet surface is larger than that in the central region (10% or less for the surface layer).

The textures of the above sample are shown in Figs. 12(a) and 12(b). The main components in the central region lie around $\{110\}\langle 112 \rangle$ and $\{211\}\langle 111 \rangle$, while those near sheet surfaces are located around $\{100\}\langle 011 \rangle$ to $\{211\}\langle 011 \rangle$ and $\{332\}\langle 113 \rangle$. The former is typical of the cold-rolling texture of fcc metal with medium S.F.E.,³³⁾ and the latter is known as the surface shear texture that develops in surface layers of fcc materials when they are subjected to heavy shear deformation during rolling.^{36,37)} Therefore, the textures clearly indicate that the model alloy has undergone heavy deformation without intense recrystallization, and a large influence of shear stresses due to the friction between roll and sample surfaces on the deformation is manifest in the surface region. For comparison, the texture of the same alloy that has been air cooled after a similar hot rolling to promote recrystallization is shown in Figs. 12(c) and 12(d); the finishing temperature of this sample is 900°C. By recrystallization, the abovementioned texture components greatly diminished, but relatively weak components appeared around $\{100\}\langle 001 \rangle$ for the central region and around $\{310\}\langle 133 \rangle$ and $\{211\}\langle 012 \rangle$ for the surface region instead.

5. Deduced Textures of Prior γ by MODF

The two samples of steel A hot-rolled and rapidly cooled to around 650°C with different Δt 's of 0.05 and 0.3 s were chosen for the MODF analysis. These samples are the same as those in Figs. 4(a), 4(b), 4(d) and 4(e). As shown in Fig.

Fig. 12. Textures of (a and b) Ni-30%Fe alloy shown in Fig. 10 and (c and d) the same alloy recrystallized by slow cooling.

Fig. 13. Textures of the steel A hot-rolled and rapidly cooled to about 650°C at Δt of 0.05 s, 0.3 s. (Upper) $\phi_2=0^\circ$ and (lower) $\phi_2=45^\circ$.

Fig. 14. Textures of γ before the transformation to α deduced by MODF using the textures of the steel A shown in Fig. 13. (Upper) $\phi_2=0^\circ$ and (lower) $\phi_2=45^\circ$.

13, the measured textures of α do not qualitatively change by changing Δt , whereas severity changes particularly near sheet surfaces. The major components of the measured textures are always located around $\{110\}\langle 112 \rangle$ and $\{211\}\langle 111 \rangle$ near the sheet surface and around $\{100\}\langle 011 \rangle$ to $\{311\}\langle 011 \rangle$ and $\{332\}\langle 113 \rangle$ in the central region. Therefore, although some change in the texture of prior γ by the changing in Δt is expected because of the observed variation in severity,

the details cannot be understood from the measured textures of α .

Hence MODF analysis was performed to deduce the textures of prior γ before transformation. The results are shown in Fig. 14. Near the sheet surface, the texture of prior γ has been found to markedly alter by the change in Δt (see Figs. 14(a) and 14(c)). At Δt of 0.05 s, the main components lie around $\{100\}\langle 011 \rangle$ to $\{211\}\langle 011 \rangle$ as well as

around the $\{111\}$ fiber located at Φ of 54.7° in the $\phi_2=45^\circ$ section, which is very similar to the surface shear texture observed in the deformed Ni–Fe model alloy (see Fig. 12(a)). However, at Δt of 0.3 s, the $\{100\}\langle 011\rangle$ component completely disappears and the component around $\{310\}\langle 133\rangle$, which has been observed near sheet surfaces in the recrystallized Ni–Fe model alloy (see Fig. 12(c)), appears. A broad $\{111\}$ fiber is also seen in the $\phi_2=45^\circ$ section, which is perhaps a remnant of deformed γ . Therefore, near the sheet surface, the structure of the prior γ is thought to be severely deformed under shear stresses at Δt of 0.05 s, while that at Δt of 0.3 s is the one that has undergone an intense recrystallization of deformed γ . The latter is in good agreement with the microstructures shown in Figs. 9(b) and 9(c).

In the central region, on the other hand, the deduced texture of prior γ does not so largely alter by changing Δt as it does near the sheet surface, as shown in Figs. 14(b) and 14(d). The major components lie around $\{110\}\langle 112\rangle$ and $\{211\}\langle 111\rangle$ irrespective of Δt , although the minor component of $\{100\}\langle 001\rangle$ increases to some extent as Δt increases from 0.05 to 0.3 s. These major components are those typical of cold-rolled fcc alloys³³⁾ and deformed Ni–30%Fe in the central region (see Fig. 12(b)), while the $\{100\}\langle 001\rangle$ component is the one that develops during recrystallization as seen in Fig. 12(d). Thus the prior γ in the central region is deduced to be deformed at Δt of 0.05 s and to undergo slight recrystallization during the period of 0.3 s after hot rolling. These results well agree with the observation of quenched structures shown in Figs. 9(d) to 9(f).

6. Discussion

The most interesting phenomenon observed in this study is that the reduction in Δt as well as that in rolling pass interval has remarkably reduced the grain size of α in plain C–Mn steel. The grain size, in particular, near sheet surfaces decreased from 2.5 to 1.3 μm by the reduction in Δt from 0.5 to 0.05 s alone, and it further decreased to below 1 μm by reducing both Δt and rolling pass interval together. Here we discuss the mechanism of this extreme grain refinement.

The aspects of the grain refinement mechanism to be first discussed are (1) whether the structure of γ before the phase transformation to α causing the extreme grain refinement is deformed or recrystallized and (2) whether the phase transformation is static or dynamic. As to the aspect (1), the MODF analysis shown in Fig. 14 clearly suggests that the γ before the phase transformation should be deformed both in the surface and central regions, since the deduced textures of prior γ at Δt of 0.05 s exhibit the representative characters of fcc shear texture or rolling texture. The elongated shape of γ grains observed in the quenched samples in Figs. 9(d) to 9(f) also indicates the same mechanism operating in the central region.

In respect to the aspect (2), the microstructures shown in Figs. 9(d) to 9(f) also suggest that the phase transformation in the central region should be static, since the structures in the central region after hot rolling have been able to be quenched into martensite. In the region near the sheet surface, the relevant results to the aspect (2) are the deduced

texture of prior γ and the microstructures of quenched samples at Δt around 0.3 s (see Figs. 9(b), 9(c) and 14(c)). These data evidently show that the structure 0.3 s after hot rolling is a recrystallized γ with the grain size of about 5 μm . Therefore, in order to explain this result by assuming that the phase transformation is dynamic, the structure has to have undergone a reverse transformation from α to γ and then a grain growth of γ to the grain size of 5 μm for the period of 0.3 s, after the dynamic transformation from γ to α . This elaborate process is, however, unlikely to occur within such a short period and around A_{e3} , although Yada and his coworkers have reported that the reverse transformation may occur after DSIT around A_{e3} .³⁸⁾ Moreover, this process is unable to explain the deduced texture of prior γ shown in Fig. 14(c). The deduced texture shows the distinct characters of recrystallization from ‘deformed γ ’, since it consists of the recrystallization components such as $\{310\}\langle 133\rangle$ and the diffuse $\{111\}$ fiber that is most likely to be a remnant of deformed γ as mentioned earlier. If the structure underwent the above elaborate process involving the two transformations ($\gamma \rightarrow \alpha \rightarrow \gamma$) and the growth after the dynamic transformation, its texture would be largely different from the recrystallization texture shown in Fig. 14(c). Thus dynamic transformation is in contradiction to the results in this study. In contrast, the mechanism based on static transformation can simply explain these results by recrystallization of deformed γ after hot rolling. It can be therefore concluded that the phase transformation leading to the pronounced grain refinement in this study should be static, and the transformation is the one from deformed γ both in the surface and central regions.

Since, in the surface region, a large amount of shear strain is superimposed on the plane strain that is the only component imparted in the central region, it would increase the number of nucleation sites of α as well as the rate of recrystallization of deformed γ . The finer grain size and the faster kinetics of recrystallization observed in this region are mainly ascribed to this shear strain. The reduction in Δt effectively suppresses such accelerated recrystallization as well as recovery of deformed γ , so that the dramatic grain refinement may take place owing to the increased number of nucleation sites by the presence of shear strain. In the central region where such shear strain is absent, slower recovery and recrystallization are suppressed, and then it causes a less pronounced grain refinement as shown in Fig. 3.

Similarly, in SSMR, the additional reduction of the last pass interval perhaps suppresses the recovery and recrystallization of γ that otherwise occur during the interval, and therefore even finer grain structures as shown in Fig. 7 can appear due to the enhanced accumulation of strain. It is believed that the basic mechanism of grain refinement in SSMR is the same as that described above, since, although grain size is finer, the grain structures as well as the textures observed for SSMR are very similar to those observed by the reduction in Δt alone. Note that, since the smallest pass interval attained by SSMR, 0.17 s, is close to the time at which the recrystallization of deformed γ becomes intense near sheet surfaces (see Fig. 14(c)), the suppression of recrystallization during the pass interval under the present SSMR conditions might only be partial near the sheet surface.

In such a heavily deformed or work-hardened γ , the increase in nucleation sites for α has been discussed in relation with increase in high angle grain boundary areas due to elongated grain shape in the rolling direction, increased nucleation potency at the grain boundaries by the introduction of grain boundary ledges, and additional nucleation sites due to deformation substructures.³⁹⁾ It is naturally expected that all the three mechanisms operate in the formation of the present grain refinement. Grain elongation, grain boundary ledges and deformation substructures are seen in Figs. 10 and 11. With respect to the deformation substructure, Hurley *et al.*³⁵⁾ examined the microstructure of hot-rolled Ni–30%Fe in their study on DSIT and reported that the inter-granular cell walls that had misorientation angles ranging from 1 to 20° might play a primary role for the nucleation sites in DSIT. Similar cell walls are seen in the present results (see Figs. 10 and 11). Therefore the grain refinement in this study may also be ascribed to these cell walls, of which misorientation angle has ranged from 1 to over 15° as seen in Fig. 10. However, it is still indefinite that the low angle cell walls with misorientation angle less than 15° can contribute as largely to the increase in nucleation sites as high angle boundaries. A contrasting implication in this study is that the MODF analysis, in which about 70% of α is assumed to nucleate on “high angle γ boundaries” under the double KS relation (*i.e.* $\omega=0.7$), has remarkably well deduced the texture of γ before transformation; no other variant selection model proposed so far is able to deduce the texture of prior γ so reasonably as the present model. Furthermore, it is noteworthy that the averaged spacing between high angle boundaries (more than 15° in misorientation) seen in Fig. 11 in the direction normal to the rolling plane (the smallest spacing direction) is about 1.2 and 2.2 μm for the surface and central regions respectively, which are 1 to 1.5 times the grain sizes in the corresponding regions for the ultra-fine α shown in Figs. 4 and 7. Therefore if the nucleation potency at high angle grain boundaries is enough enhanced by the ledge mechanism, such distribution of high angle boundaries as observed in the model Ni–Fe alloy may be able to provide the most of nucleation sites necessary for the grain refinement in this experiment. In this scenario of grain refinement, the presence of low angle boundaries (*e.g.*, less than 10° of misorientation) might provide it with additional nucleation sites as assumed in the present MODF analysis; about 30% of α may precipitate on low angle boundaries with ω being equal to 0.7. Nevertheless further study is obviously required to thoroughly elucidate such a form of nucleation sites to cause the extreme grain refinement in this experiment.

7. Conclusions

A new approach to breakthrough in grain refinement of C–Mn steel has been described, which may allow us to manufacture hot-rolled steel sheets consisting of polygonal α of about 1 μm in grain diameter with reduced difficulty, and the grain refinement mechanisms have been discussed based on several aspects of experimental result and the calculation of textures of prior γ by MODF. The following conclusions have been drawn in this study.

(1) As plain C–Mn steel is hot-rolled above A_{e3} , a dramatic effect in grain refinement appears by reducing the interval between the finish of rolling and the start of rapid cooling, Δt . Near sheet surfaces, the grain size of α gradually decreases from about 2.5 to 2 μm with decreasing Δt from 0.5 to 0.2 s, and then a sudden drop to 1.3 μm occurs as Δt is reduced to 0.05 s. In thickness center regions, the grain size gradually decreases from over 3 μm to about 2 μm , as Δt is reduced from 0.5 to 0.05 s.

(2) A further grain refinement is achieved by the reduction in rolling pass interval between the last two passes along with the abovementioned reduction in Δt . As the pass interval is decreased from 0.6 to 0.17 s, the grain size is decreased to 0.87, 1.1 and 1.4 μm near the sheet surface, a quarter of sheet thickness below the surface and in the central region respectively. Consequently, the steel sheets, in which the ultrafine grain structure of about 1 μm in grain diameter penetrates from the sheet surface to the depth of a quarter of the sheet thickness, are obtained.

(3) The ultrafine grains near the sheet surface are remarkably equiaxed, while those in the central region are slightly elongated in the rolling direction. They are mostly surrounded by high angle boundaries, and fine cementite particles are present on the grain boundaries near sheet surfaces.

(4) The transformation causing the present grain refinement is considered to be static (during post-rolling cooling) and from an unrecrystallized deformed γ . Owing to the reduction in Δt and the pass interval, the accumulation of strain in the deformed γ is enhanced, and a thereby increased number of nucleation sites for α lead to the extreme grain refinement.

(5) The smaller grain size near the sheet surface is mainly ascribed to the presence of shear strain due to the friction between roll and sample surfaces. The calculated texture of prior γ by MODF in this region well agrees with a shear type of texture observed in Ni–30%Fe model alloy. Because of the shear strain, the kinetics of recrystallization of γ is accelerated, but since this accelerated recrystallization as well as the recovery of dislocations is effectively suppressed by the reduction in Δt and the rolling pass interval, the grain refinement to below 1 μm is achieved. In the central region where such shear strain is absent, the slower recrystallization and recovery of dislocations are suppressed and then it causes less pronounced grain refinement.

(7) In the Ni–Fe model alloy, elongated grains in rolling direction, small equiaxed grains surrounded by high angle boundaries as well as inter-granular low angle boundaries that form equiaxed cellular structures are observed. The average diameter of the cells is about 0.4 μm in the surface region and the averaged spacings between high angle grain boundaries in the direction normal to the rolling plane are 1.2 and 2.2 μm in the surface and central regions respectively, which are 1 to 1.5 times the grain sizes of the ultra-fine α obtained in this study.

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