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Effect of the Second-Phase Particle Precipitation on the Recrystallization Texture of High-Strength and Fine-Grain Interstitial-Free Steel

Abstract

© 2020, Springer Science+Business Media, LLC, part of Springer Nature. The effect of the second-phase particle precipitation on the recrystallization texture of high-strength and fine-grain interstitial-free steel was studied. It is shown experimentally that the sizes of second-phase particles increase and their number decreases with holding time and annealing temperature. The texture is mainly influenced by the size, number, and distribution of the particles. Finely dispersed second-phase particles are strongly pinned on the grain boundaries, which significantly hinders the development of {111} surface textures. It the pinning force on the grain boundary decreases, the {111} surface texture improves due to the aggregation and growth of second-phase particles with holding time. The strength of the γ -phase ({111} texture) grows and reaches maximum (annealing temperature 850°C), finally declines with annealing temperature. Precipitated particles of 40–60 nm favor the development of the {111} texture component.

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Effect of the Second-Phase Particle Precipitation on the Recrystallization Texture of High-Strength and Fine-Grain Interstitial-Free Steel

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The effect of the second-phase particle precipitation on the recrystallization texture of high-strength and fine-grain interstitial-free steel was studied. It is shown experimentally that the sizes of second-phase particles increase and their number decreases with annealing time and temperature. The texture is mainly influenced by the size, number, and distribution of the particles. Fine and dispersed second-phase particles are strongly pinned on the grain boundaries, which significantly hinders the development of {111} surface textures. As the pinning force on the grain boundary decreases, the {111} surface texture improves due to the aggregation and growth of second-phase particles with annealing time. The strength of the γ -phase ({111}<112> texture) grows and reaches maximum (annealing temperature 850°C), finally declines with annealing temperature. Precipitated particles of 40–60 nm favor the development of the {111} texture component.

Keywords: second-phase particles, precipitation, recrystallization texture, grain boundary, high-strength and fine-grain interstitial-free steel.

Introduction. With the rapid development of the automobile manufacturing industry, energy saving and emission reducing become more and more important. How to reduce the weight of the automobile body under the condition of safety become a very important issue [1–4].

It is well known that interstitial-free steel (IF steel) is a kind of deep-drawing steel which is widely used in the manufacture of automobile body panels because of its good drawability, surface quality, and recycling [5–7]. The drawability of IF steels, usually quantified in terms of the Lankford value r , is strongly dependent on recrystallization texture. IF deep drawing steel has good drawability because of its strong recrystallization texture γ fiber (<111> //ND) and weaker α fiber texture (<110> //ND). Plastic strain ratio (r value) is the main index influencing IF steel's forming performance, and high r value indicates that the steel plate has a good forming performance [8–10].

A new IF steel added the Nb element was developed in this study and the strengthening of dispersion and grain refinement can be achieved by Nb(C, N) precipitates. Excellent deep drawability (high r -value) can be obtained by fixing C and N, and high strength steel can be obtained by grain refinement of steel [11–15].

The recrystallization texture of IF steel is influenced by many factors, such as the chemical composition, steelmaking, hot rolling, cold rolling, and annealing process. Many researchers had different views about the effect of the second-phase particles on recrystallization texture. Some researchers believed that the formation of large and homogeneous second-phase particles can improve the recrystallization textures obtained during the process of cold rolling and recrystallization annealing. Other researchers, however, claimed that the presence of second-phase particles during cold rolling have no effect on the recrystallization textures [16–18].

In recent years, the effect of the precipitation behavior of the second-phase particles on recrystallization texture was studied. Wang et al. [19] found that the second-phase particles of TiS and Ti(C, N) gather and grow during the process of hot rolling, as well as the fine FeTiP particles precipitated at the same time. Zhao et al. [20] studied the precipitation behavior of the second-phase particles in Ti-bearing steel and found that when the amount of Ti added to the steel is very small, the interstitial atom N will combine with Al to form the second-phase particles. Song et al. [21] had discovered the evolution of

microstructure and texture of 0.2%C–Mn steel during large warm deformation and subsequent annealing. It was reported that the removal of C from the solid solution by the precipitates can benefit to $\{111\}$ texture in IF steel [22, 23]. Ghosh et al. [24, 25] presented an overview of the different types of precipitates in high strength interstitial free steels and reported that the formability decreases with the increase of the volume fraction of FeTiP precipitation.

The effects of the second-phase particles precipitation on mechanical properties and the recrystallization texture of IF steel have been studied in recent years, however, there are very little research into the effect of second-phase particles precipitation on the formation mechanism of the recrystallization texture until now.

The second-phase particle precipitation and its effect on the recrystallization textures of the fine grain and high-strength IF steel were studied in this paper. The morphology and composition of the second-phase particles and the intensity change of the γ -fiber texture ($\{111\}<112>$) under different annealing processes were also investigated.

1. Experimental Materials and Methods.

1.1. Experimental Materials. The experimental steel was melted in the laboratory of University of Science and Technology Liaoning. Nb was added to the experimental steel in order to form fine and dispersed second-phase precipitates. The chemical compositions of experimental steel are shown in Table 1.

Table 1

Chemical Compositions of Experimental Steel (wt.%)

Element	C	Si	Mn	P	S	Nb	B
Content	0.0043 ± 0.002	0.267 ± 0.04	1.88 ± 0.04	0.062	0.0063	0.097 ± 0.03	0.0007

1.2. Experimental Methods.

1.2.1. Experimental Procedure. Sample dimension experimental steel was $200 \times 100 \times 1$ mm ($L \times W \times H$). Cold rolling was conducted by the cold rolling mill in the State Key Laboratory of Rolling and Automation, Northeastern University. Cold rolling reduction was 85%. The annealing experiments were carried out in the annealing furnace at the University of Science and Technology of Liaoning. Experimental steel were heated to 830, 850, and 870°C, respectively, and annealing time were 5, 10, 20, 40, and 60 min, respectively, under an annealing temperature of 850°C after cold rolling.

The primary TEM samples were taken based on the metallographic experiment, and the surface of samples eroded with 4% nitric acid alcohol solution was treated by sprayed carbon, and then the samples with carbon membrane were put into a 4% nitric acid alcohol solution until the carbon membrane separated from the samples. The carbon membranes were taken out by the copper net and put on the filter paper. Finally, TEM samples were obtained.

Annealing process of experimental steel is shown in Table 2.

Table 2

Annealing Process of Experimental Steel

Number	Heating rate, °C/s	Temperature, °C	Annealing time, min	Cooling type
1	5	830	40	Air-cooled
2	5	850	5, 10, 20, 40, 60	
3	5	870	40	

1.2.2. Microstructure and Texture (Grain Orientation). The microstructure of the samples were observed by ZEISS optical microscope. The morphology, size, quantity, and components of the second-phase particles were obtained by extraction complex experiment using JEOL-2100 TEM and EDX. Microzone Orientation Imaging analysis were performed by the analytical testing technique of JSM6480LV – SEM + PEGASUS2040 – EDS + EBSD. The OIM5.31 (Orientation Imaging Microscopy) software was used for texture analysis. The phase of experimental steel was obtained by XRD.

2. Results and Discussion.

2.1. Experimental Results under Different Annealing Temperatures.

2.1.1. The Effect of Second-Phase Particles on the Texture of Experimental Steel under Different Annealing Temperatures. The morphology and EDX of the second-phase particles and orientation density function (ODF) figures under different annealing temperatures (annealing time is 40 min) are shown in Fig. 1.

It can be seen that when the annealing temperature is at 830°C, a large number of fine second-phase particles are distributed uniformly in the matrix as shown in the left side of Fig. 1a. The size of the large particle with the irregular rectangular shape confirmed NbCN by EDX as shown in the right side of Fig. 1a is about 50 nm (circled with red line), and the sizes of small particles with the round or oval shape are about 20 nm. When the annealing temperature is at 850°C, the number of precipitation particles decrease and the particles size increases. It can be seen that the small particles gather together. As a result, the number of the particles decrease and the size of second-phase particles increases with the increase of the annealing temperature. At the same time, the average grain size is about 50 nm with round shape as shown in the left side of Fig. 1b. When the annealing temperature is at 870°C, the rectangular shape particle (circled with red line) is confirmed as Nb(CN) by EDX as shown in the right side of Fig. 1c.

2.1.2. Two Phase Particle Parameters and ODF Data at Different Annealing Temperatures. The size of two-phase particles was calculated using Photoshop and IPP. SEM+EBSD was used for texture analysis, and Channel 7 software was used for ODF analysis. ODF data in different annealing temperatures were obtained by EBSD. It can be seen that the intensity value of the strongest point (close to $\{111\}<112>$) of γ fiber texture in experimental steel changes greatly with the increase of annealing temperature. The values are 10.0, 14.6, and 13.1, respectively, as shown in the right side of Fig. 2 and Table 3. When the annealing temperature is at 830°C, a large number of NbC particles with the size of 10 to 30 nm are distributed in the matrix. The precipitated particles are pinning on the grain boundary. As a result, the grain growth is hindered. Consequently, the recrystallization texture cannot form sufficiently and the development of the $\{111\}$ texture component is significantly restrained, and the texture intensity value is only 10. With the increase of the annealing temperature from 830 to 850°C, the precipitated second-phase particles in the steel become large and uniform, with an average size of 60 nm. The pinning force of sparse and coarse second-phase particles on grain growth during recrystallization annealing process becomes weaker, and the grain recrystallization is completed due to little impact of the sparse and coarse second-phases on ferrite grain growth. The growth rate of $\{111\}$ grain orientation is significantly higher than random grain orientation and others grain orientation after annealing as shown in Table 3, subsequently, the $\{111\}$ orientation grains can grow up completely. The intensity of the $\{111\}$ texture component increases gradually and reach to the maximum value of 14.6 at the annealing temperature of 850°C. With the decrease of the amount of large particles and the particles at 870°C the annealing temperature, the $\{111\}$ texture component is less than that of at 850°C, the annealing temperature, as shown in Fig. 1 and Table 3. Hence, it can be seen that the size and the number of second-phase particles have a great effect on the development of texture. The effects of the second-phase particles on the recrystallization texture of the high strength IF steel are mainly determined by the size, the number and the distribution of the particles as shown in Fig. 1.

Fig. 1. The morphology and EDX of second-phase and ODF figures under different annealing temperatures (annealing time is 40 min): 830 (a), 850 (b), and 870°C (c).

2.1.3. XRD Analysis of the Experimental Steel. The crystal structure and the phases formation of the the experimental steel surface at 830, 850, and 870°C annealing temperature for 40 min annealing time were investigated by the XRD analysis as shown in Fig. 2. It can be seen that the crystal structure and the phases of the experimental steel surface at 830, 850, and 870°C annealing temperature for 40 min annealing time are α (ferrite).

Table 3
Parameters of Second-Phase Particles and ODF Data in Different Annealing Temperatures

Parameter	Temperature (°C)		
	830	850	870
ODF (max)	10.0	14.6	13.1
Particles size (nm)	20	60	100
Volume fraction (%)	0.12	0.08	0.04
Component	NbC	Nb(CN)	Nb(CN)

Fig. 2. XRD analysis on the surface of the experimental steel at 830, 850, and 870°C annealing temperature.

2.2. Experimental Results under Different Annealing Times.

2.2.1. The Microstructure of Experimental Steel under Different Annealing Times.

Figure 3 shows the microstructure of the experimental steel at different annealing times. It can be found that the ferrite grains grew gradually with the increase of holding time. From 20 to 40 min, the ferrite grain was fully recrystallized, and the ferrite grain arrangement was relatively regular.

Fig. 3. Microstructure of experimental steel at 850°C under different annealing time: 5 (a), 10 (b), 20 (c), and 40 min (d).

Fig. 4. The grain size chart of 1/2 layer at 850°C under different annealing times: 5 (a) 10 (b), 20 (c), and 40 min (d).

2.2.2. The Grain Size Distribution of Experimental Steel. The statistical distributions of grain size in the 1/2 layer of experimental steel at different annealing times were measured using EBSD, as shown in Fig. 4. It can be seen that the grain size of 1/2 layer under different annealing times is mostly concentrated around 13 μm . The grain size increases with the increase of holding time.

Table 4

Mechanical Properties of Experimental Steel in Different Annealing Times

Holding time (min)	Tensile strength (MPa)	Yield strength (MPa)	Elongation (%)	<i>n</i>	<i>r</i>
5	456.0	261.4	34.1	0.23	2.03
10	407.2	225.5	34.3	0.22	1.95
20	431.4	242.0	28.0	0.21	1.88
40	447.6	246.7	36.8	0.24	2.13
60	453.2	282.4	34.1	0.23	1.91

2.2.3. Effect of Annealing Time on Mechanical Properties of Fine Grain High Strength IF Steel. Mechanical properties of experimental steel can be measured by tensile test as shown in Table 4. With the increase of annealing time, the tensile strength and yield strength changed little. The elongation decreases at first and then rises, *n* value (work hardening index) and *r* value (plastic strain ratio) were both increase, and peak value appears at 40 min annealing holding time, and then falls again.

2.2.4. The Effect of Second-Phase Particles on the Texture of Experimental Steel under Different Annealing Times. The morphology, EDX of second-phase particles and texture at different annealing times are shown in Fig. 5. It is can be seen that the precipitated second-phase particles consist of some large and small particles. The sizes of large particles are from 40 to 100 nm and the sizes of small particles are from 10 to 40 nm. When the annealing time is 20 min, the size of the large precipitated particles reaches up to 80 nm. The second-phase particle with the rectangular shape (circled with a red line) is confirmed as the carbonitride of Nb (NbCN) by EDX results as shown in the right side of Fig. 5a and its size is about 50 nm. When the annealing time is between 20 to 40 min, the small second-phase particles disappears completely and the amount of large particles decrease. The second-phase particle with the round shape (circled with a red line) is confirmed as the carbide of Nb (NbC) by EDX as shown in the right side of Fig. 5b and its size is about 50 nm. The second-phase particle with the irregular rectangle shape (circled with a red line) is confirmed as the carbonitride of Nb (NbCN) by EDX as shown in the right side of Fig. 5c and its size is about 100 nm. The second-phase particles with the round or oval shape are mainly NbC. The second-phase particles with square or irregular rectangular shape are mainly conformed as Nb (C, N).

The ODF ($= 45^\circ$) in its half layer of the experimental steel under different annealing times at 850°C is shown in the right side of Fig. 5, and it is combined with the EDX results. It is shown that all the textures of in its half layer of the experimental steel under different annealing times are the strong γ -fiber texture ($\{111\langle uvw \rangle\}$) and the texture component is the same. The γ -fiber texture of experimental steel with 40 min annealing time is the strongest, and the maximum density value is 14.6. The $\{111\}\langle 110 \rangle$ texture turns to $\{111\}\langle 112 \rangle$ texture as the annealing time is increased.

2.2.5. XRD Analysis of the Experimental Steel. The crystal structure and the phases formation in half layer of the experimental steel under 40 min annealing time was investigated by the XRD analysis as shown in Fig. 6. It can be seen that the crystal structure and the phases in half layer of the experimental steel is α (ferrite).

2.2.6. Orientation Imaging Analysis of the Experimental Steel. The orientation imaging analysis of the experimental steel in its half layer at different annealing times is shown in Fig. 7. When the orientation difference is larger than 15° , the grain boundary is called the large angle grain boundary, and the $2\text{--}15^\circ$ orientation difference is called the small angle grain boundary. The different colors represent the different grain orientations.

Fig. 5. Morphology and EDX of large second-phases and ODF figure under different annealing times: 20 (a), 40 (b), and 60 min (c).

The blue color grain shows the crystal plane of $\{111\}$ parallel to the rolled surface, the red color grain represents the $\{001\}$ crystal plane parallel to the rolled surface, while the green color grain represents the $\{101\}$ crystal plane parallel to the rolled surface.

It can be seen that the ferrite grains appear recrystallized and the grain sizes become uniform and equiaxial. When the recrystallization is completed, their grain boundaries are

Fig. 6. XRD analysis of the experimental steel in half layer under 40 min annealing time.

Fig. 7. Orientation imaging map of half layer under different annealing times.

Fig. 8. The α -orientation and γ -orientation line of half layer under different annealing times.

large angle grain boundaries and most of the grain orientations concentrate to the $\langle 111 \rangle$ and $\langle 112 \rangle$ direction. As a result, there are more blue color grains, which represent the $\{111\}$ orientation. The grains grow with the increase of annealing time. When the annealing time is 40 min, the change of texture is not obvious and the microstructure is uniform.

2.2.7. The α -Orientation and γ -Orientation Line of Half Layer under Different Annealing Times. The intensity changes of α and γ fiber textures are shown in Fig. 8 under different annealing times. The left figure represents α fiber texture, and γ fiber texture is shown in the right one. The intensity change of α fiber recrystallization texture from $\{001\}\langle 110 \rangle$ to $\{112\}\langle 110 \rangle$ is very large with the change of angle θ for experimental steel, especially for the texture of $\{001\}\langle 110 \rangle$ and $\{111\}\langle 110 \rangle$. The intensity of α fiber recrystallization textures improve when the annealing time extends from 5 to 10 min. The peak of texture intensity changes from $\{112\}\langle 110 \rangle$ to $\{111\}\langle 110 \rangle$ and the density of $\{111\}\langle 110 \rangle$ texture decreases with the increase of annealing time. The intensity of $\{111\}\langle 110 \rangle$ decreases when the annealing time changes from 5 to 20 min. The intensity changes of $\{111\}\langle 110 \rangle$ are small when the annealing time extends from 20 to 60 min. The intensity change of $\{111\}\langle 112 \rangle$ reaches to the maximum value when the annealing time is 40 min.

Conclusions

1. The sizes of second-phase particles become larger, and their numbers decrease with the increase of annealing temperature and annealing time.

2. The textures of experimental steel are mostly based on the $\{111\}$ and $\{112\}$ textures when the annealing temperatures are 830, 850, and 870°C, respectively. The intensity of the γ fiber texture ($\{111\}\langle 112 \rangle$) first increases and then subsequently reduces with the increase in the annealing temperature. When the annealing temperature is 850°C, the intensity of the $\{111\}$ surface texture of experimental steel rise up to the maximum.

3. The effect of the second-phase particles on recrystallization texture is mainly determined by the size, the number and the distribution of the particles. Fine and dispersive second-phase particles have a strong pinning force on grain boundaries, which significantly hinder the development of $\{111\}$ surface textures. The weak pinning force of coarse and sparse second-phase particles on grain boundaries promote the development of $\{111\}$ texture.

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