Formation of Mono-Layer Honeycomb Structure in High-Purity Iron by Single Pass Hot-rolling

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Microstructure change in high-purity irons was investigated at various cooling rates after single pass hot-rolling. Large columnar grains, which are named mono-layer honeycomb grains, developed through the thickness when high-purity iron (C:1.5 mass ppm, N:0.8 mass ppm, S:1.5 mass ppm, Si:1.5 mass ppm, Mn:0.29 mass ppm) was hot-rolled at 1273 K and cooled in a furnace with the average cooling rate of about 8.5 $\times$ 10$^{-3}$ K/s. Mono-layer honeycomb grains developed in neither high-purity iron water-quenched after hot-rolling, nor middle-purity (C: 1.5 mass ppm, N: 9 mass ppm, S: 10 mass ppm, Si+Mn+P: 42 mass ppm) and low-purity (C: 20 mass ppm, N: 8 mass ppm, Si+Mn+P: 270 mass ppm) irons. The crystal orientation of mono-layer honeycomb grains was determined by electron back scattering pattern method. The crystal orientation of mono-layer honeycomb grains does not have any specific $\alpha$-texture. Grain growth behavior was investigated by using the cold-rolled and annealed irons. The grains in the high-purity iron grew faster and larger than those in middle-purity and low-purity irons. It is considered that the hot-rolled $\gamma$-grains in the high-purity iron recrystallize and coarsen in cooling, and then the $\gamma$-grains transform into $\alpha$-grains. The $\alpha$-grains grow larger due to the effect of purification. Consequently, mono-layer honeycomb grains are developed in high-purity iron.

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1. Introduction

Many researchers have studied the microstructure and texture of hot-rolled carbon steels and low carbon steels.\textsuperscript{1-10} They have mainly focused on the formation of transformation textures. Kubo\text{\textit{dera}}\textsuperscript{1} investigated the microstructure and texture of carbon steels containing 0.05 mass% C and 0.29 mass% Mn after hot-rolling in the $\gamma$-phase region. It was cleared that the homogeneous grains developed and the main and sub textures of $\alpha$-phase were $\{100\}0\{11\}$ and $\{110\}0\{11\}$, respectively. The formation of such textures is explained by assuming the variant selection. The $\gamma$-texture obtained after hot-rolling was assumed to be $\{110\}0\{112\}$ and $\{112\}0\{11\}$, and $\alpha$-texture was calculated according to the Kudrjumow-Sachs [K-S] relationship.\textsuperscript{2} Jones\textsuperscript{3} reported that the measured $\{100\}0\{11\}$ texture of carbon steels containing 0.1 mass% C, 0.8 mass% Mn and 0.03 mass% Nb corresponds to the calculated $\alpha$-texture, after assuming 24 variants according to the K-S relationship act with equal possibility.

The specimens tested in the previous studies contained several hundreds or thousands mass ppm of carbon, nitrogen, manganese and niobium. Abiko\textsuperscript{11} showed that the microstructure, recrystallization and transformation behavior of ultra-purified iron (C: 1.5 mass ppm, N: 0.8 mass ppm, S: 1.5 mass ppm, Si+Mn+P < 1.5 mass ppm) after hot-rolling were quite different from those of conventional steels. Namely, columnar grains developed along the normal direction, when the samples were hot-rolled in the $\gamma$-phase region and cooled in air with the average cooling rate of about 3.1 K/s. The development of columnar grains depended on the purity of iron. Especially, in high-purity iron, giant columnar grains grew from the surface to the center in thickness. Ogawa\textsuperscript{12} investigated the microstructures of high-purity irons (C: 1.1 mass ppm, N: 2.1 mass ppm, S: 0.73 mass ppm, Si+Mn+P < 1.5 mass ppm) hot-forged at various temperatures. Giant columnar grains developed through the thickness in parallel to the forging direction when the high-purity iron was forged at 1263 K and cooled in the strew-ash with the average cooling rate of about 0.1 K/s. However, the formation of large columnar grains thorough the thickness under hot-rolling is not clear in detail. The main purpose of the present study is to investigate the microstructure and the texture development of the high-purity iron after single pass hot-rolling in the $\gamma$-phase region.

2. Experimental Procedure

2.1 Sample preparation

Three kinds of irons (HP, MP and LP irons) were prepared. The chemical compositions of irons are shown in Table 1. The high-purity iron ingot was made of high-purity electrolytic irons of more than 99.995 mass% purity by the furnace with a copper cold-crucible.\textsuperscript{13} The middle-purity iron and the low-purity iron ingots were melted in ceramics crucibles. The high purity iron ingot was heated at 1425 K and was hot-forged to the sheet bar of 20 mm in thickness. The middle-purity iron ingot and the low-purity iron ingot were heated at 1425 K and were hot-rolled to the sheet bars of 25 mm in thickness. These iron sheets were heated to 1373 K and hot-rolled to the sheets of 5.0–5.3 mm in thickness by 3 passes. The hot-rolled iron

\begin{table}[h]
\centering
\caption{Chemical compositions of irons tested (mass ppm)}
\begin{tabular}{cccccccc}
\hline
Irons & C & N & O & S & Si & Mn & P \\
\hline
HP & 1.5 & 0.8 & 74 & 1.5 & 0.2 & <0.2 & 0.4 \\
MP & 10 & 9 & 80 & 10 & 20 & 20 & 2 \\
LP & 20 & 8 & 86 & 10 & 70 & 100 & 100 \\
\hline
\end{tabular}
\end{table}
sheets were machined into 100 mm length, 40 mm width and 4 mm thickness and used for following experiments.

2.2 Single pass hot-rolling
The specimens were heated again at 1373 K for 1.8 ks, and then single pass hot-rolling was conducted at 1273 K with the hot-rolling speed of 1.67 m/s. Thickness of the high-purity (HP) iron after hot-rolling was 1.5–1.7 mm, and thickness of the middle-purity (MP) and the low-purity (LP) irons were 2.1–2.3 mm. One specimen was water-quenched just after hot-rolling, and the other was put into a furnace kept at 1123 K and cooled to 573 K in furnace. The average cooling rate was about \( \frac{8.5 \times 10^{-3}}{} \) K/s. Microstructures and crystal orientations of each sample were investigated. Crystallographic orientation measurement for the transversal direction (TD) plane of the HP iron was carried out by an electron back scattering diffraction pattern (EBSD) method. 14)

2.3 Recrystallization and Grain growth behavior
The specimens of 4 mm in thickness were cold-rolled to the sheets of 1 mm in thickness. The sheets were annealed at various temperatures for 60 s before water-quenching. Grain size of each specimen was measured by point-counting method.

3. Experimental Results

3.1 Microstructure after single pass hot-rolling
Figure 1 shows the microstructures of the TD plane of the HP iron cooled in furnace and water-quenched after hot-rolling. Each microstructure corresponds to the whole cross section along the hot-rolling direction: the top and bottom side showed the rolled surface of each sample. The microstructure evolved under furnace cooling (Fig. 1(a)) is mainly constructed of giant columnar grains, which will be called mono-layer honeycomb grains in this paper. The microstructure water-quenched after hot-rolling has some columnar grains. However, the mono-layer honeycomb structure does not exist in Fig. 1(b).

Figure 2 shows the microstructures of MP and LP irons cooled in furnace after hot-rolling. Mono-layer honeycomb grains are not observed in Fig. 2.

3.2 Texture analysis of HP iron
Figure 3 shows crystal orientations of twenty-one mono-layer honeycomb grains in the HP iron cooled in furnace. Crystal orientations of each grain were plotted on (001) pole figure as solid circle marks. It does not seem that any kinds of specific texture develop. We will mention about double circle marks later.

3.3 Recrystallization and Grain growth behavior
Figure 4 shows the relationship between the annealing temperatures and grain size of each sample. The grain size became larger as the annealing temperature increased. The average grain size of the HP iron annealed at 1123 K reached over 150 \( \mu m \) which was about one and half times as large as that of the MP iron, and about four times as large as that of the LP iron.

4. Discussion
Mono-layer honeycomb structure develops in the HP iron hot-rolled at 1273 K and slowly cooled in furnace, as shown in Fig. 1(a). We will discuss (1) \( \gamma \)-texture after hot-rolling, (2) \( \gamma \rightarrow \alpha \) transformation, and (3) grain growth in \( \alpha \)-region.

4.1 \( \gamma \) texture after single pass hot-rolling
In this study, it is considered that the hot-rolled \( \gamma \)-grains in the HP iron recrystallize and coarsen because they were cooled in furnace after hot-rolling at 1273 K. It is difficult to determine the crystal orientation of \( \gamma \)-grains after hot-rolling. However, it is well known that \( \alpha \)-phase in carbon steels can be transformed from \( \gamma \)-phase according to the K-S relationship. 15,16) We assumed \{001\}(100) textures as recrystallized texture in \( \gamma \)-phase, 7,17) and then calculated the crystal orientation relationship between \( \alpha \)-phase and \( \gamma \)-phase according to the K-S relationship; 1,11,16)

\[ (111)_{\gamma} \parallel (011)_{\alpha} \text{ and } [10\bar{1}]_{\gamma} \parallel [1\bar{1}1]_{\alpha} \]

Figure 3 shows the comparison between the measured crystal orientation of twenty-one honeycomb grains in the HP iron (solid circle) and the calculated \( \alpha \)-texture (double circles). Most of measured crystal orientations disagree with the calculated texture. The crystal orientations of \( \gamma \)-grains in the HP iron after hot-rolling could not be determined in this study.

4.2 Velocity of \( \alpha/\gamma \) interface and grain growth
Abiko and Sadamori showed, from a direct in-situ observation of the \( \alpha \rightarrow \gamma \) transformation of irons using a high-temperature optical microscope, 18) that the velocity of mobile
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Fig. 2 Microstructures in TD plane of (a) MP and (b) LP irons cooled in furnace after hot-rolling.

Fig. 3 Crystal orientations of mono-layer honeycomb grains and calculated $\alpha$-texture.

Fig. 4 Relationship between annealing temperatures and grain size of HP, MP, and LP irons.

$\alpha/\gamma$ interface of high-purity iron containing 2 mass ppm carbon is $3 \times 10^{-3}$ m/s. On the other hand, the velocity of $\alpha/\gamma$ interface of the low-purity iron containing 24 mass ppm carbon is $0.5 \times 10^{-3}$ m/s. The velocity of $\alpha/\gamma$ interface during the $\gamma \rightarrow \alpha$ transformation in HP irons might be very fast due to low contents of solute impurities and segregated impurities in grain boundaries.

The grains in the cold-rolled and annealed HP irons have grown faster and larger than those in MP and LP irons (Fig. 4). $\alpha$-grains in the HP iron after hot-rolling and transforming is expected to grow larger.

4.3 Formation of mono-layer honeycomb grain in HP iron

It is considered that $\gamma$-grains in the HP iron recrystallize and coarsen while they are hot-rolled and cooled in furnace. The coarsened $\gamma$-grains transform into $\alpha$-grains as temperature goes down. Some $\alpha$-grains can grow until those grain size become larger than thickness of the HP iron due to the effect of purification. Therefore, mono-layer honeycomb grains can develop in the HP iron.

5. Conclusions

Microstructures and crystal orientations of irons after single pass hot-rolling have been investigated. The results obtained are summarized as follows.

(1) Large columnar grains, which are named mono-layer honeycomb grains, develop through the thickness in the HP iron cooled in furnace after single pass hot-rolling.

(2) The crystal orientations of mono-layer honeycomb grains do not have any specific texture.

(3) $\alpha$-grains in the HP iron have grown faster and larger than those in MP and LP irons.

(4) It is considered that the hot-rolled $\gamma$-grains in high-purity iron recrystallize and coarsen in cooling, and then the $\gamma$-grains transform into $\alpha$-grains. The $\alpha$-grains grow faster and larger due to the effect of purification. Consequently, mono-layer honeycomb grains develop in high-purity iron.
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