Grain Size Distribution Law of Cold-rolled Low-carbon Steel with Various Holding Temperatures

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Abstract—With the cold-rolled low-carbon steel sheet produced by CSP process as the object of study, uniformity of microstructure and grain size distribution law of “pancake” (elongated) grains were investigated. The samples were annealed under laboratory condition to simulate batch annealing processes with holding temperatures from 660 °C to 700 °C. Conclusions are drawn that the grain size distribution of annealed test steel along both the rolling direction and the thickness direction of the sheet samples accords well with the law described by the Log-normal function.

1. INTRODUCTION
Grain refinement strengthening is the principal mechanism that causes the strength of CSP (compact strip production) hot-rolled low-carbon sheets to be higher than that of conventional hot-rolled low-carbon sheets [1]. As cold-rolling feeds, because of high yield strength, the energy consumption in cold-rolling increases and the minimum thickness after rolling decreases. Besides, refinement of hot-rolled sheets causes the rate of recrystallization nucleation to increase and the grains in the annealed sheets to be refined as well, and leads to high yield strength and bad formable performance. As a result, cold-rolled low-carbon sheets produced by CSP technology are unsuitable to be applied to cases with higher requirements in deep-drawing [2].

Research on cold-rolled sheets produced by CSP technology is still at an initial stage, mainly on coarsening ferrite grains of annealed sheets to lower yield strength, but less study on uniformity of microstructure of this material. For research on grain structures, a basic method is to calculate the average radius of grains and the standard deviation, but this method does not reflect the whole characterization of grain structure. Therefore, grain size distribution law is introduced to study the uniformity of microstructure and regularity of grain size distribution, and has been widely used on copper processed by equal channel angular pressing [3], nanocrystalline Nd2Fe14B magnets [4] and ultrafine-grained low carbon steel [5], but not on regular low-carbon ferrite steels with strong anisotropy on grain structure.

The objective of the present investigation is to elucidate the grain size distribution law of cold-rolled low-carbon steel sheets produced by CSP technology. Three theoretical functions were introduced in this study for comparing with the test results. In groups of annealing experiments, by varying holding temperatures (660°C, 680°C, 700°C), the effects of annealing parameters on grain structure and grain size distribution law were manifested as well. Main factors and their effects on the grain size distribution law of test steels are discussed in this paper.
2. EXPERIMENTAL DETAILS
The test steel was obtained after CSP hot rolling. The thin slabs were casted to 70 mm in thickness, re-heated at 1020 °C and hot rolled to 3.5 mm in thickness with a finishing temperature of 880 °C and coiled at 550 °C. The hot-rolled sheets were cold rolled to 71.4% reduction to get 1.0 mm in thickness. All the cold-rolled sheets were cut into 10 mm sample pieces. The annealing experiments were carried out in furnace with argon atmosphere, and the heating rate was 35°C/h, and holding temperatures were 660°C, 680°C and 700°C respectively with holding time 10 hours. All the annealing experiments were ended by furnace-cooling for 3 hours then water quenched. In this paper, to study the grain size distribution laws of “pancake” grain structure, the side surface of test steel sheets was taken as microstructure observation surface. The chemical composition of the test steels produced by the CSP process is presented in Table 1.

| TABLE 1. CHEMICAL COMPOSITION OF THE TEST STEEL (WT. %) |
|----------------|----------------|----------------|----------------|----------------|
| C   | Si   | Mn   | P   | S   | Als |
| 0.043 | 0.021 | 0.15 | 0.012 | 0.004 | 0.022 |

Since the size of single grain is difficult to be measured directly due to the irregular shapes of grains in annealed test steel. In this study, the statistics of intercepts was adopted to substitute for that of grain size. The approach was to arrange a plurality of equally-spaced straight lines in parallel in a field of view vertically and laterally separately, and an interval between two points between the same grains on the line was chosen as an intercept. The measured vertical sheet intercept was designated as \( l_t \), while that along the rolling direction as \( l_r \). After measurement, the intercept averages in the two directions (\( l_t-a \) and \( l_r-a \)) were calculated. With the acquired data being divided into groups at even intervals, the number of intercepts was designated as \( N_t \) and \( N_r \). Their maximums \( N_t-m \) and \( N_r-m \) were solved, and \( N_r/N_t-m \) and \( N_t/N_t-m \) were used to generalize the intercept numbers in each group. Here individual intercepts were not counted or analyzed because they do not represent the actual size of a grain, instead, \( l_t/l_t-a \) and \( l_r/l_r-a \) were used as statistical samples so that the statistical results can reflect the law of actual grain size distribution. In order to guarantee the universality and accuracy of measurement results, the annealing experiments were repeated for three times, and three samples were chosen for each experiment, and ten fields of view were measured randomly for each sample.

3. RESULTS AND ANALYSIS
3.1. Microstructures
Microstructures of annealed test steels with different holding temperatures are shown in Fig.1. It can be seen that ferrite grains of test steels are still having an obviously “pancake” type feature, and when the heating process is same, holding temperatures has a certain impact on grain structures. In the test steel annealed at 660 °C, there are still many small equiaxed grains as well as significantly slender grains, but the “pancake” grains in the test steel annealed at 680 °C and 700 °C is more coarsening, especially in the thickness direction, and the number of small equiaxed grains decrease.

![Figure 1. Optical micrographs of annealed test steel with different annealing temperatures (a) 660°C (b) 680°C (c) 700°C](image-url)
3.2. Grain size distribution law
Grain size distribution has generally been applied to study the uniformity of microstructure. To investigate the grain size distribution law of the test steel, three theoretical functions are used in this paper: Louat function [6], Log-normal function and Weibull function [7], as given in (1)-(3) respectively, and determining coefficient R² of the fitness are calculated to reflect the degree of coincidence.

\[ f(x) = 2ax \exp(-ax^2) \]  
\[ f(x) = \frac{1}{\sqrt{2\pi \sigma x}} \exp\left(-\frac{(\ln x - \mu)^2}{2\sigma^2}\right) \]  
\[ f(x) = \frac{\beta}{\alpha^\beta x^{\beta-1}} \exp\left(-\frac{x}{\alpha}\right) \]

![Grain size distribution of test steel with different holding temperatures in rolling direction: (a) 660°C, (c) 680°C, (e) 700°C; in thickness direction: (b) 660°C, (d) 680°C, (f) 700°C](image)

Figure 2. Grain size distribution of test steel with different holding temperatures in rolling direction: (a) 660°C, (c) 680°C, (e) 700°C; in thickness direction: (b) 660°C, (d) 680°C, (f) 700°C

The measured grain size distributions of the test steel with different holding temperatures compared with the three functions are shown in Fig. 2, the parameters of these functions are summarized in Table
2. It is clearly in Fig. 2 that the grain size distribution of test steel along both the rolling direction and the thickness direction fit the log-normal function fairly well.

| Direction | Holding temperature (°C) | Weibull | Log-normal | Louat |
|-----------|--------------------------|---------|------------|-------|
|           | β                       | α       | σ         | μ     | R²    | a | R² |
| Thickness | 660                      | 2.1133  | 1.1030    | 0.8164| 0.5413| -0.0095| 0.9247| 0.8064| 0.8003|
|           | 680                      | 2.4238  | 1.0515    | 0.9080| 0.4666| -0.0543| 0.9408| 0.8979| 0.8670|
|           | 700                      | 1.8671  | 1.0969    | 0.8395| 0.6043| -0.0310| 0.9209| 0.8474| 0.8505|
| Rolling   | 660                      | 1.7006  | 1.0052    | 0.8962| 0.7454| -0.1876| 0.9295| 1.0495| 0.8743|
|           | 680                      | 1.7708  | 0.9699    | 0.8193| 0.6308| -0.1981| 0.9019| 1.1089| 0.8152|
|           | 700                      | 1.9035  | 1.0055    | 0.9029| 0.6316| -0.1805| 0.9409| 1.0005| 0.8985|

4. DISCUSSIONS AND CONCLUSIONS
For annealed cold-rolled low-carbon steel sheet produced by CSP technology, ferrite grains are mainly in “pancake” sharp. The samples were annealed under laboratory condition to simulate the batch annealing process with holding temperatures from 660 °C to 700 °C. In present experiments, grain size distribution of annealed test steel fits the Log-normal function better than the Weibull function and the Louat function.

Most workers agreed that the “pancake” grain structure after recrystallization results from the reduced nucleation frequency and the extended growth range of the new grains, combined with the tendency for AlN precipitation to occur in “sheets” on prior grain boundaries and sub boundaries extended in the rolling plane. These sheets may provide anisotropic deformation-cell structure could itself promote growth along the rolling direction [8].

In summary, many factors affect uniformity of microstructure as well as grain size distribution law of annealed cold-rolled low-carbon steel sheets produced by CSP process, some of these factors hamper the migration of grain boundaries, such as AlN precipitation, and some speed up the movement of grain boundaries, like size difference between adjacent grains increases, and some are still complex, for instance, the CSL boundaries.

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