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Effect of annealing parameter on microstructure and magnetic properties of cold rolled non-oriented electrical steel

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Abstract: The microstructure and magnetic properties of cold rolled non-oriented electrical steel, annealed at 200−1 000 ℃ for 0−240 min with different heating rates, were investigated by optical microscopy, scanning electron microscopy, Epstein frame, and transmission electron microscopy. The results show that the magnetic properties of cold rolled non-oriented electrical steel can be improved by controlling the annealing process to obtain uniform coarse grains with critical sizes after the recovery, recrystallization and growth of grains. Additionally, the annealing temperature influences the magnetic properties more significantly than annealing time, and with the increase of heating-up rate during the annealing process, the magnetic properties of the cold rolled non-oriented electrical steel increase.

Key words: non-oriented electrical steel; annealing process; magnetic property

1 Introduction

Cold rolled non-oriented electrical steels, having good magnetic and workability properties, play an important part in electric machine, rectifier, and electric transformer. It is a new kind of soft magnetic material. Currently, Japan and Germany hold the key technology to produce stable products[1]. With the combination of domestic markets with aboard markets, the markets for the energy saving electrical machines are huge. At present, researchers have tried many ways to enhance the magnetic properties by materials design, cast process control, dispersed precipitate control, hot/cold roll process, heat treatment and so on. Obviously, annealing parameter of heat treatment affects microstructure and magnetic properties of rolled non-oriented electrical steel effectively[2].

In this work, influences of annealing temperature, annealing time and heat-up rate on the microstructure and magnetic properties of cold rolled non-oriented electrical steels were discussed.

2 Experimental

The cold rolled non-oriented electrical steel samples were provided by Hunan Valin Lianyuan Iron and Steel Company. The compositions (mass fraction, %) of the samples are listed in Table 1.

The steel plates were cut into the size of 300 mm× 30 mm, and the amounts of the sample perpendicular to the rolling direction and parallel to the rolling direction were the same. The annealing experiments were carried out in the self-made high-temperature tube resistance furnace. The annealing temperature was 0−1 000 ℃, annealing time was 0−240 min, heating rate was 0−100 ℃/min, and the temperature fluctuation was 1 ℃. The mixture of 65% H_2 and 35% N_2 was applied as protective atmosphere during the annealing process and the current of air velocity was 3 L/min.

The microstructure of the samples after different heat treatment processes were observed on the Polyvar-MET optical microscope after corrupted by sweet spirit of 3% aqua fortis and grain size was analysed

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Table 1 Chemistry composition of cold rolled non-oriented electrical steel samples

C	Si	Mn	P
0.03−0.04	0.29−0.35	0.43−0.48	0.03
S	Als	Sb	Fe
0.003	0.10−0.12	0.02−0.23	Bal.

by Q550 quantitative metallographic analyser. The magnetic properties were measured by Epstein frame method. The amounts of the samples perpendicular to the rolling direction and parallel to the rolling direction were the same and the samples were jointed to a square. The total mass was approximately 1 kg. The magnetic loss ($P_{15/50}$) was measured under the condition of 1.5 T and 50 Hz. The magnetic induction density was measured in magnetic field intensity of 5 kA/m. The microstructures of the samples were characterized by KYKY-Amray 2008B scanning electron microscopy and Philips TecnaiG220 transmission electron microscopy.

3 Results and discussion

3.1 Microstructure and magnetic property

The optical microstructure of cold rolled non-oriented electrical steels is shown in Fig.1. The microstructure of the cold rolled samples consists mainly of refined and uniform equiaxed grains, and no typical cold rolled fibrous tissues are observed. It might be due to the fact that the cold rolling process is carried out at the higher temperature but not at room temperature, and this phenomenon that temperature rises can be caused by deforming heat during the rolling process[3].

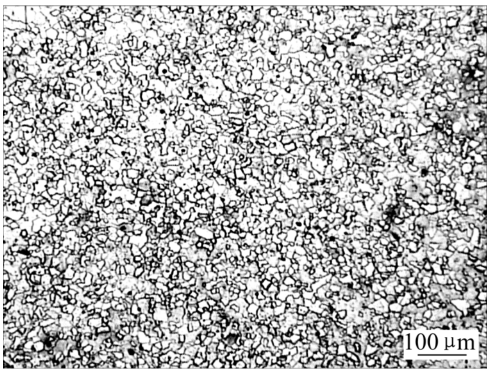

Fig.1 Microstructure of cold rolled non-oriented electrical steel

There are some non-metallic inclusions in the cold rolled sheet, as shown in Fig.2. However, amount of the inclusions in the steels is very small and their size is also rather small. This indicates that the steel is very clean and can be treated as having little inclusions. Therefore, the cold rolled non-oriented electrical steel exhibits magnetic loss of 16.36 W/kg and intensity of magnetic induction of 1.70 T.

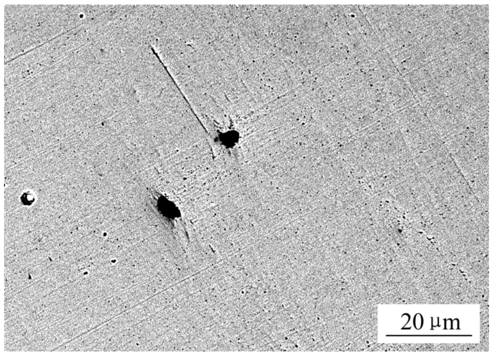

Fig.2 Non-metallic-inclusion of as-rolled non-oriented electrical steel

3.2 Influence of annealing temperature on hardness, microstructures and magnetic properties

The microstructures of the rolled plates after annealing for 60 min at different temperatures are shown in Fig.3. In Fig.3(a), the material is in the recovery stage and the rolled plates still keep fine equiaxed crystal grain. Novel grains appear in the cold rolled plate with the increase of annealing temperature, meanwhile, the recrystallization begins and the growth of some grains is observed, forming fine uniformly distributed recrystallization grains, as show in Fig.3(b). When the annealing temperature continues to increase, grains grow rapidly, forming uniformed coarsening structure, as shown in Fig.3(c).

The magnetic properties of rolling plates versus temperature annealed at different temperatures for 60 min are shown in Fig.4. When the plates are in low temperature recovery stage, both grain size and magnetic properties vary slightly. Then, with the increase of temperature, recrystallization takes place in the plate, meanwhile, the magnetic properties decrease rapidly. When temperature is up to 900 ℃, the plate is in a coarse grain uniformly distributed state and the magnetic properties steady descend slowly. The magnetic property reaches the nadir at 950 ℃ and the mean grain size is 63 μm. When the temperature is higher than 950 ℃, the hysteresis loss descends slightly with the increase of temperature. The paramount parameter that influences the magnetic property is grain size if other factors are almost the same, including plate's thickness, variety of the impurities, crystal orientations, internal stress and its distribution[4]. When the grains grow, the grain boundary circumstance will decrease. The decrease of the grain boundary circumstance results in the descending of the hysteresis loss (P_h). The relationship

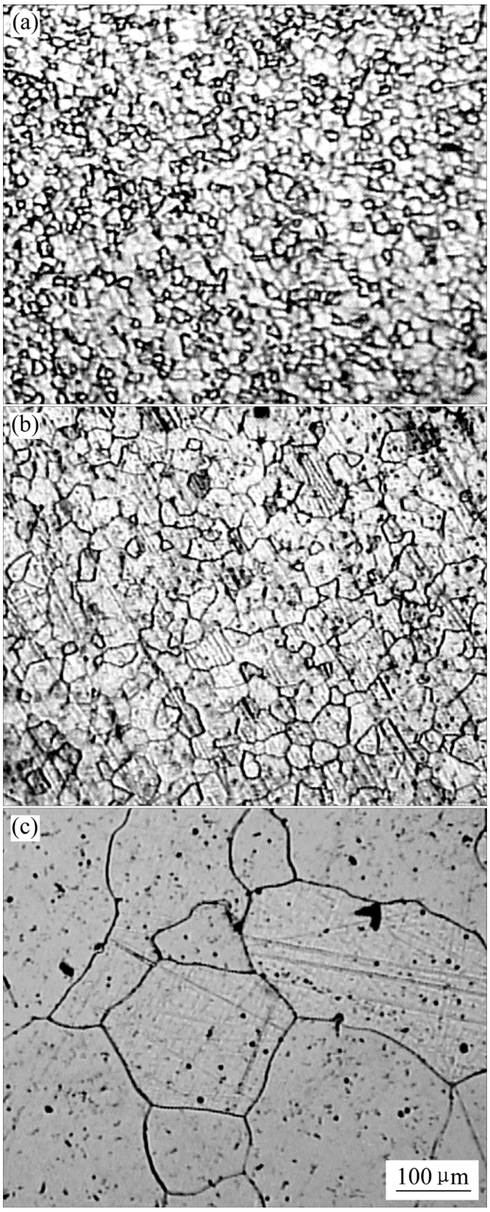

Fig.3 Microstructures of steel annealed for 60 min at different temperatures: (a) 200 ℃; (b) 500 ℃; (c) 800 ℃

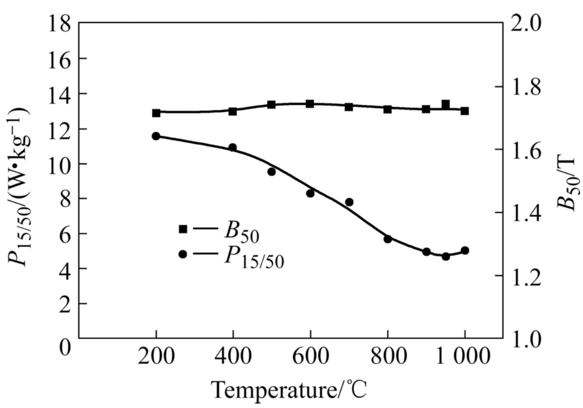

Fig.4 Influence of annealing temperature on magnetic properties of rolled plates annealed for 60 min

between the grain size and the domain walls width is given as follow:

$$d^{3/4}=\lg(\frac{\gamma}{K_1})^{(\delta/1.32)} \quad (1)$$

where γ is the domain wall energy in a unit domain area; K_1 is the magneto crystalline anisotropy constant and δ is the domain width. The domain width increases with the increase of the grain size, resulting in the increase of eddy current loss[5]. As a result, a critical grain size exists to decrease the iron loss. Ref.[6] reported that when the content of silicon was less than 1%, the critical grain size of non-oriented electrical steel approximately was equal to 50−80 μm. The results are conformed by the experiment results in this study. The magnetic induction strength B_{50} only varies slightly with the increase of the annealing temperature. The influence of grain size on magnetic induction is complex. If only concerning the baffle magnetization effect of grain boundary, the magnetic induction strength will increase with the grain growth. In fact, crystallographic texture is also one of main (factors) parameters that influence the magnetic induction strength[7−8], and more investigation is needed in this area.

3.3 Influence of annealing time on microstructure and magnetic property

For the rolled steel heated at 700 ℃ and 950 ℃ for a serial of time, respectively, the magnetic loss–time curve is shown in Fig.5. This clearly indicates that when the rolled steel plates are heated at 700 ℃ continuously, with the extension of annealing time, magnetic loss descends slowly. The magnetic loss reaches stable status after annealing for 60 min and it only decreases slightly with the extension of time after 60 min. Whereas, when the rolled steel plates are heated at 950 ℃, the magnetic loss reaches a stable status only after 15 min and it only decreases slightly with the extension of time after 15 min. Under the same annealing time, the iron loss of the rolled

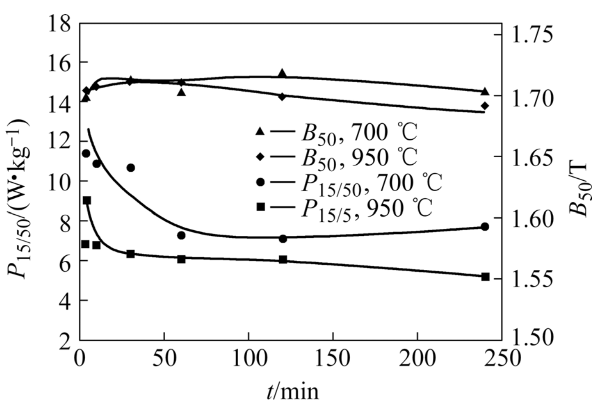

Fig.5 Influences of annealing time on magnetic properties of rolled plates annealed at 700 ℃ and 950 ℃

plates annealed at 950 ℃ is lower than that of the rolled plates annealed at 700 ℃. However, the induction density of rolled plates annealed at 700 ℃ is higher than that of the rolled plates annealed at 950 ℃. The induction density only varies slightly with the extension of annealing time. The microstructures of the rolled plates after annealing at 700 ℃ and 950 ℃ are shown in Fig.6. After annealing for a long time, such as annealing for 240 min, recrystallization takes place and the grain size is larger than the initial grain size, as shown in Fig.6(a). Whereas, after annealing at 950 ℃ for 4 min, as shown in Fig.6(b), large grains annex small grains and the mean grain size is larger than that of the rolled plate annealed at 700 ℃ for a long time. This is due to the fact that when annealing at high temperature, the driving force acquired for the secondary recrystallization is reduced and the grains are apt to grow rapidly. The grains in rolled plates after annealing at 950 ℃ for a long time, such as 240 min, are almost uniformly large grains, as shown in Figs.6(c) and 7(b). The grain boundaries are flat, indicating that recrystallization takes place and no precipitated phase accumulates on the grain boundary. The uniformly, dispersed and refined precipitated phase existed in the matrix can reduce the activation energy, promote the recrystallization and inhibit the excessive growth of grains[9], then the uniformly coarse grains are obtained, as shown in Fig.7(b).

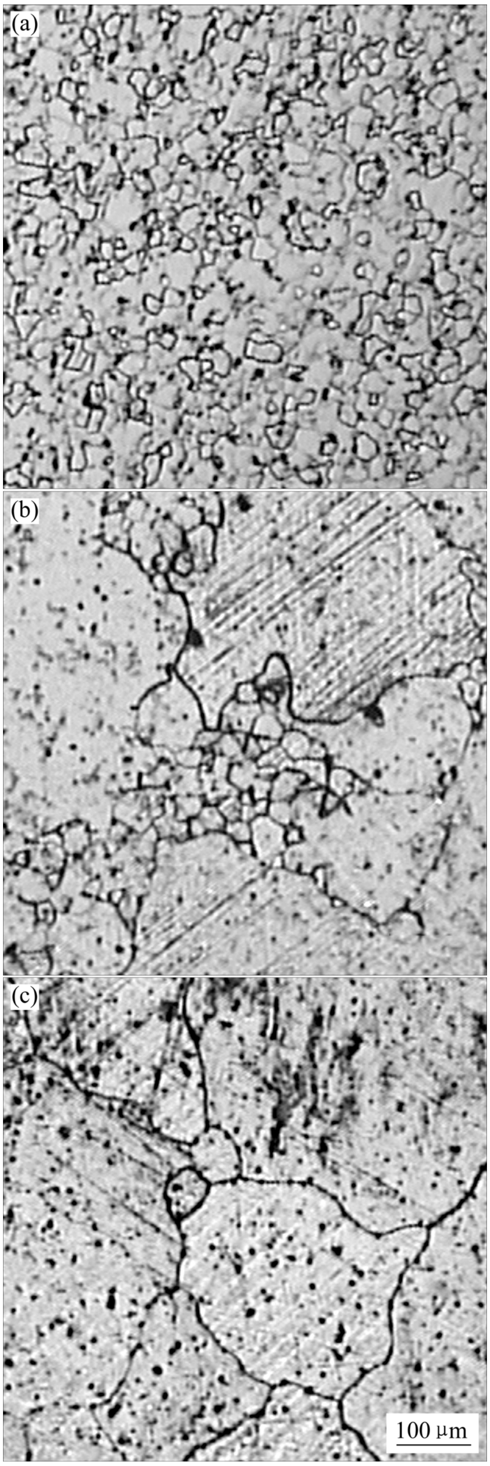

Fig.6 Microstructures of steel heated at 700 ℃ and 950 ℃ for different conditions: (a) 700 ℃, 240 min; (b) 950 ℃, 4 min; (c) 950 ℃, 240 min

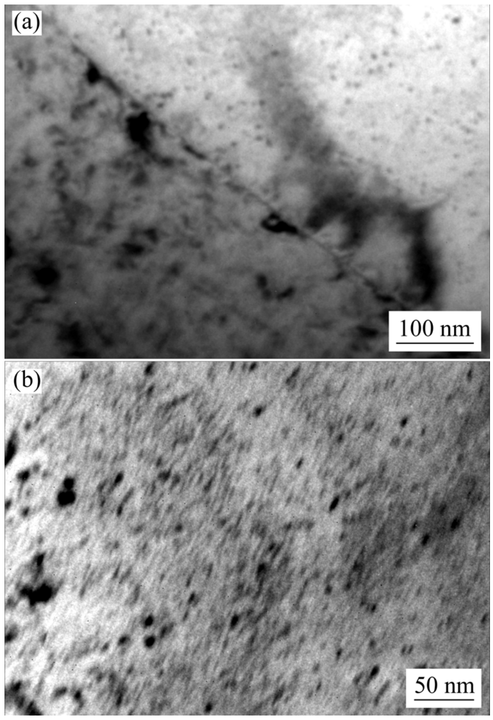

Fig.7 TEM micrographs of rolled electrical plates annealed at 950 ℃

3.4 Influence of heating rate on steel's microstructure and magnetic property

The variations of the magnetic properties of the rolled plates after annealing at 950 ℃ for 60 min with different heat-up rates are listed in Table 2.

The induction density increases and the iron loss decreases with the increase of the heat-up rate. With the increase of the heat-up rate, the grain sizes get larger, as

Table 2 Influence of heating up rate on steel's magnetic property

Heating-up rate/(℃·min^{-1})	P_{15}/(W·kg^{-1})	B_{50}/T
30	5.24	1.74
73	4.91	1.75
100	4.79	1.75

shown in Fig.8. When the heat-up rate increase rapidly, the stored energy cannot be released in such a short time.

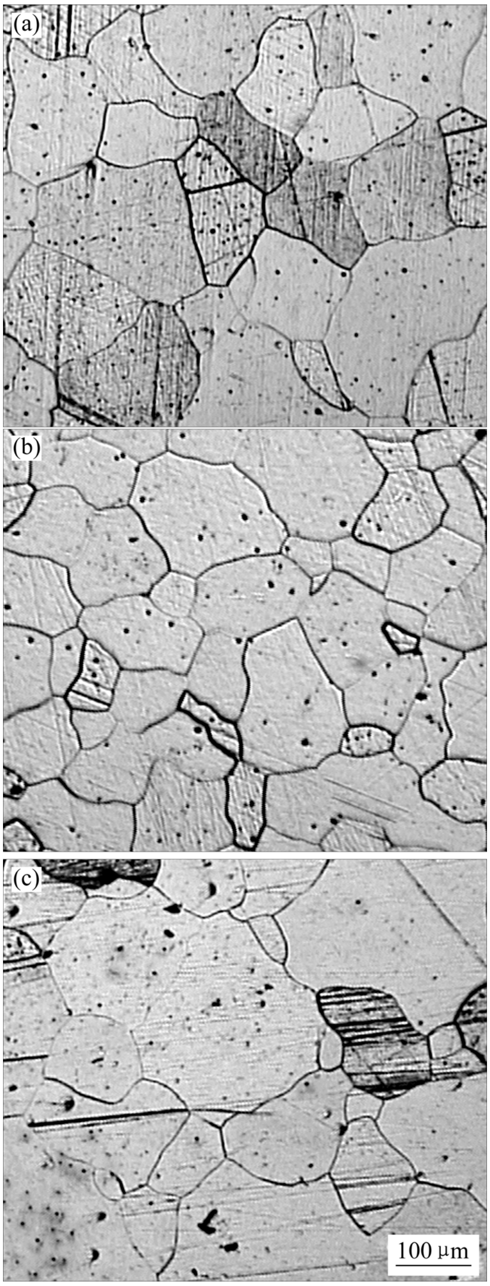

Fig.8 Microstructures of steel heated at 950 ℃ for 60 min at different heating rates: (a) 30 ℃/min; (b) 73 ℃/min; (c) 100 ℃/min

There is adequate driving force for the recrystallization process, which is beneficial to the recrystallization and the grain growth[9]. Additionally, it is also helpful for ameliorating the texture, resulting in the improvement of the magnetic properties[10].

4 Conclusions

1) With the increase of annealing temperature and extension of annealing time, recovery, recrystallization and grain growth take place in the rolled plates. The magnetic properties improve along with the growth of the grains. A critical grain size exists, which can make the iron loss least.

2) The annealing temperature influences the magnetic properties more than the annealing time.

3) The magnetic properties are improved with the increase of the heat-up rate.

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