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Effect of copper precipitates on the stability of microstructures and magnetic properties of non-oriented electrical steels

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A R T I C L E I N F O

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A B S T R A C T

Non-oriented electrical steels with different amounts of copper were prepared and the microstructure and magnetic properties of each kind of steel were studied. The results show that there exist a large number of Cu-rich metastable precipitates in the hot-rolled bands of the steels containing copper. They not only can decrease the sensitivity of the microstructures and magnetic properties of the steels to the change of process parameters but also can significantly reduce the core loss of the steels by improving the recrystallization textures without obviously decreasing the magnetic induction. Therefore, it is possible to control the microstructures and then magnetic properties of non-oriented electrical steels by the copper precipitates.

1. Introduction

Non-oriented electrical steels have been widely used as core materials in motors and generators, in which high magnetic induction and low core loss are required [1]. The two properties are very sensitive to the process parameters and thus the production process of non-oriented electrical steels is strictly controlled. It is very important to decrease the sensitivity of the microstructures and magnetic properties of non-oriented electrical steels to the change of process parameters. However, very few investigations on this subject have been reported so far.

The precipitates in non-oriented electrical steels are usually considered to be harmful to the magnetic properties of the steels because they are generally non-ferromagnetic and hinder grain growth during recrystallization annealing [2–5]. However, recent studies show that copper precipitates can improve the magnetic properties of non-oriented electrical steels [6]. In order to further investigate the role of copper precipitates in non-oriented electrical steels, small quantities of copper were added to non-oriented electrical steels to produce a metastable Cu-rich phase. The effect of copper precipitates on the sensitivity of the microstructures and magnetic properties of the steels to the change of process parameters were investigated.

2. Materials and experimental procedures

The materials used for this study are three types of silicon steels with different levels of copper; the chemical composition of each is listed in Table 1. It can be seen that the steel A is substantially free of copper, while the steel C contains more copper than the steel B. The qualified steels were melted in a vacuum induction furnace for forging and were then hot-rolled out in 3-mm thick bands by three passes in the temperature range from 960 to 1150 °C. The temperature at which austenite begins to transform to ferrite during cooling is about 948 °C. To simulate the coiling conditions, the hot-rolled bands were maintained at 550 and 650 °C respectively for 1 h in a box resistance furnace, followed by air-cooling (henceforth referred to as simulated-coiling annealing). After a normalizing annealing at 850 °C for 1 h and at 925 °C for 21 min, all of the bands were uniformly cold-rolled to a 0.5-mm thickness and were subjected to a recrystallization annealing at 950 °C for 5 min in an atmosphere of 25% H_2+75% N_2.

After simulated-coiling annealing, normalizing annealing and recrystallization annealing, the microstructures in longitudinal cross section of the investigated steels were examined by an optical microscope. The grain sizes were measured by the standard linear intercept method. In each case, the number of grains measured at random was more than 100. The average grain sizes were estimated from such measurements.

To quantitatively determine the average size and volume fraction of the precipitates, cuboid samples of 10 mm × 10 mm × 3 mm in size were cut from the simulated-coiling annealed bands

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Table 1
Chemical compositions of the investigated steels (wt%).

Steel	C	Si+Al	Cu	Mn	S	P
A	0.0030	1.93	0.0052	0.59	0.0040	0.0062
B	0.0044	1.87	0.12	0.62	0.0006	0.0079
C	0.0025	1.93	0.25	0.62	0.0018	0.0086

and were etched in a solution containing 4 ml nitric acid and 96 ml ethanol. These samples were then examined in a field emission scanning electron microscope (SEM) supplemented by energy dispersive spectrometry (EDS). Micrographs of the precipitates were randomly taken, and the parameters mentioned above were determined using Image-Pro Plus software.

Rectangular samples with 300 mm in length and 30 mm in width were prepared to measure the magnetic properties by a single-strip tester.

3. Results and discussion

3.1. Precipitates in the hot-rolled bands

It is currently well known that fine Cu-rich phases can precipitate during the thermal aging of ferritic steels containing copper. The full transformation sequence for copper precipitation in Fe–Cu alloys is given by bcc→9R→3R→fcc with increasing aging time [7–10]. In addition, it is also reported that Cu-rich phases can precipitate in hot-rolled bands of non-oriented electrical steels containing copper and these precipitates are likely to have a B2 structure with a fully coherent relationship to the ferrite matrix [6]. Fig. 1 shows the morphology and distribution of the precipitates in the investigated steels after simulated-coiling annealing at 650 °C for 1 h. It can be seen that very few precipitates exist in the steel A (see Fig. 1a) and a large number of fine precipitates appear in the steels B and C (see Fig. 1b and c). The amount of the precipitates increases with copper content. A single particle is approximately spherical, but with increasing copper content particle coalescence occurs, which makes some precipitates look like worm (see Fig. 1c). The EDS spectra indicate that the copper level in these precipitates (3.10 wt%) is much higher than that in the matrix (0.08 wt%) (see Fig. 1d and e), which implies that the precipitates should contain Cu-rich clusters/phases based on previously reported results [6–10]. Table 2 lists the volume fractions and average size of the precipitates in the investigated steels simulated-coiling annealed at different temperatures for 1 h. It can be seen that the volume fraction of the precipitates in the steel C is significantly greater than that in the steel B and the average size of the precipitates in the steel C is also slightly larger than that in the steel B due to the increased copper content.

3.2. Optical microstructures of the investigated steels

3.2.1. Simulated-coiling annealed bands

Fig. 2 shows the optical microstructures in longitudinal cross section of the hot-rolled bands after simulated-coiling annealing at 650 °C for 1 h. It can be observed from Fig. 2a that the coarse and ribbon-like grains which were elongated along the rolling direction exist in the center layer of the hot rolled band. On both sides of the ribbon-like grains are the equiaxed grains with sizes gradually increasing from the surface to the interior because the surface layer suffers a larger deformation than the center layer in the hot rolling process and thus the nucleation sites for ferrites in the surface layer are more than that in the center layer. The deformed austenite grains in the center layer are not recrystallized during finish rolling in the nonrecrystallization austenite region. In the process of subsequent phase transformation, some ferrite grains formed, grew and became elongated grain in these deformed austenite grains due to poor nucleation capacity. This observation is in agreement with the previous reported result [11]. However, it can be seen from Fig. 2b and c that the equiaxed grains with an average size of about 30 μm uniformly distributed over the whole longitudinal cross sections and no distinct difference in the grain size can be seen from the surface to the center. There is also not any ribbon-like grain in the center layer of the hot rolled bands. This is because a large number of copper precipitates in the steels B and C increase their resistance to deformation during hot rolling and thereby make the deformation more uniform throughout the thickness of the hot rolled bands. Moreover, the fine and dispersed precipitates strongly impede the growth of the ferrite grains by pinning the grain boundaries and therefore, the grains in steels B and C are smaller than that in steel A. The optical microstructures of the hot-rolled bands simulated-coiling annealed at 550 °C for 1 h are similar to those described above.

3.2.2. Normalizing annealed bands

After normalizing annealing, the optical microstructures in longitudinal cross section of the hot rolled bands simulated-coiling annealed at the two temperatures are basically the same and the typical microstructures are given in Fig. 3. It can be observed that the ribbon-like grains in the steel A have been recrystallized (see Fig. 3a and b) and the abnormal growth of some grains occurred in the hot rolled band simulated-coiling annealed at 650 °C for 1 h and normalizing annealed at 850 °C for 1 h (Fig. 3a) and in the hot rolled band simulated-coiling annealed at 550 °C for 1 h and normalizing annealed at 925 °C for 21 min. Grains in the steels B and C grow obviously, but their sizes are still substantially uniform (see Fig. 3c and d). Table 3 lists the average grain size of each hot rolled band after normalizing annealing. It can be seen that the average grain size of the steel A is significantly affected by the process parameters, whereas that of the steel C is less influenced by the process parameters. The steel C has a smaller average grain size than the steel B.

Tanaka et al. [12] reported that the coincidence site lattice (CSL) boundaries have usually lower grain boundary energies and higher migration mobility than general grain boundaries in non-oriented electrical steels, especially when impurity atoms segregate at grain boundaries. Gomes et al [13] reported that the surface grains with ‹100›// ND orientation tend to rotate 15° along the Euler angle Φ to match {115} fiber which exhibits a Σ3 CSL relationship with both {110} and {111} fibers. The last two fibers are commonly found in non-oriented electrical steels. Thus, during normalizing annealing, the abnormal growth of some grains in the steel A is likely to be associated with the grains having CSL boundaries with adjacent grains and requires a proper combination of the process parameters of simulated-coiling annealing and normalizing annealing because as mentioned above, abnormal grain growth occurred only in two hot rolled bands of the steel A.

On the other hand, the copper precipitates in the steels B and C can inhibit the migration of CSL boundaries and impede abnormal grain growth during normalizing annealing, making the grain sizes more uniform. With the increasing number of precipitates, the resistance to the migration of grain boundaries also increases. Hence, the average grain size of the steel C is smaller than that of the steel B.

3.2.3. Final recrystallized sheets

Fig. 4 shows the typical optical microstructures of the final recrystallized sheets. It can be observed that the recrystallized grains in the steel B are larger than that in the steels A and C, and