Effect of Rare Earth Element La on Texture and Inclusion of Non-oriented Electrical Steel Produced by Thin Slab Casting and Rolling Process

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Effect of rare earth element La on non-oriented electrical steel produced by thin slab casting and rolling process is investigated by using transmission electron microscope, scanning electron microscope and X-ray diffraction. The results show that, (1) The columnar crystals are refined obviously with the addition of rare earth element La, the primary dendrite arm spacing is reduced from 2.42 mm to 1.76 mm, and the secondary dendrite arm spacing is reduced from 0.35 mm to 0.28 mm. (2) The banded structure of the hot rolled plate in center is significantly improved after adding rare earth, the thickness of the banded structure is reduced from 0.75 mm to 0.24 mm. (3) With the addition of rare earth element La, the precipitation morphology of AlN transforms from lump to spherical, the density and amount of inclusions are greatly reduced. (4) The average grain size of finished product is increased with the addition of rare earth element La, meanwhile, the unfavorable texture of (111) decreases and the favorable texture of (100) and (110) increases. With the addition of rare earth, the average core loss is decreased by 0.4 W/kg, and the average magnetic induction intensity is increased by 0.011T.

KEY WORDS: element La; non-oriented electrical steel; texture; inclusion; CSP.

1. Introduction

Non-oriented electrical steels are widely used as rotor materials for electrical machines such as motors and generators, which is an important indicator of the level in special steel development.1–5) Thin slab casting and rolling process is the high-efficiency and compact process, which has been researched by domestic and foreign scholars.6–8) Italy Terni Company made use of thin slab casting and rolling process to produce electrical steel in 2000, Ma Steel has realized the industrialized production of non-oriented electrical steel by the compact strip production (CSP) process in 2005.

The magnetic properties of non-oriented electrical steel include magnetic induction intensity and iron loss, which are mainly affected by chemical composition, impurities, inclusions, grain size and texture.9,10) The inclusions in steel not only inhibit grain growth, cause lattice distortion, but also block magnetic domain and domain wall motion, and deteriorate the magnetic energy of the steel. Therefore, the inclusions should be reduced as far as possible.1,11,12) The magnetic properties of non-oriented electrical steel are mainly depended on the development of steel making process, in which the chemistry and cleanliness are directly controlled, and also the development of the calcium-treatment is to reduce the negative effect of inclusions.1,13–17) But the content of oxygen and sulfur can not be reduced by calcium-treatment, and the inclusion morphology of MnS and AlN can not be well controlled. The rare earth treatment technology has been established to be one of the most promising methods for controlling inclusions, which will replace the calcium-treatment in the future.11,18,19) However, most studies of the electrical steel production with rare earth are going to focus on traditional process, and the references to thin slab casting and rolling process technology are few.

In the present work, a trial production of non-oriented electrical steel was made in CSP production line, the role of rare earth in the non-oriented electrical steel produced by thin slab casting and rolling process was studied. The purpose of this work is to understand the influence of rare earth on microstructure, texture and precipitates of non-oriented electrical steel.

2. Experiment

The non-oriented electrical steel with rare earth was produced by CSP production line, 50W600 was used as the target grade, and the contrast tests were carried out. Table 1 shows the chemical composition of the test steel.

The non-oriented electrical steel produced by 180 Ton Converter Automation Smelting, RH refining and CSP. Rare
earth was added at the end of RH alloying, the long nozzle and argon gas were used to prevent the increase of nitrogen and oxygen during the continuous casting process. The temperature of slab into the tunnel furnace was above 950°C, the slab was reheated to 1150°C, hot rolled to 2.5 mm with the final rolling temperature and the coiling temperature were 920°C and 800°C, respectively. After pickling, the hot rolled plate was cold rolled to 0.5 mm with 80% reduction and subjected to annealing at 850°C for 3 min. The process was the same except the addition of rare earth. The magnetic properties of the finished product are shown in Table 2. The average iron loss is decreased by 0.4 W/Kg, while the average magnetic properties are increased by 0.011 T after adding rare earth.

The growth of columnar crystals and equiaxed crystals were observed after the slab section was corroded. The hot rolled plate, the cold rolled plate and the finished product were processed into metallographic samples with the size of 20 mm (rolling direction) × 15 mm (transverse direction) × thickness, etched with a 4% nital and examined using an optical microscope (OM). The size distribution of the precipitates was determined using German ZEISS EVO18 scanning electron microscope (SEM) and American FEI Tecnai G2 F20 transmission electron microscopy (TEM), the samples were electropolished by perchloric acid after grinded, then removed the film by perchloric acid after the carbon was sprayed. The textures were measured based on Holland PANalytical X'Pert PRO X-ray diffraction across the thickness, the orientation distribution functions (ODFs) and the component of texture components were calculated from three incomplete pole figures {110}, {200} and {211}.

3. Results and Discussion

3.1. Effect of Rare Earth Treatment on Microstructure Evolution

3.1.1. Effect of Element La on Slab Macrostructure

As shown in Fig. 1, the columnar crystals of the slab are coarse and even transgranular without adding rare earth element La, but those refine and fragment after adding rare earth element La, the proportion of the equiaxed crystals is increased. It is found that the primary dendrite arm spacing of the slab is decreased from 2.42 mm to 1.76 mm after adding rare earth element La.

The secondary dendrites are analyzed in order to illustrate the refining degree of rare earth. As shown in Fig. 2, the secondary dendrite arm spacing of slab 1# and 2# are 0.35 mm and 0.28 mm respectively, and the secondary dendrite arm spacing is reduced by 0.07 mm after adding rare earth element La.

3.1.2. Microstructure of the Hot Rolled Plate

According to contrast microstructures with adding rare earth and without adding rare earth, the surface of the hot rolled plate is recrystallization microstructure, there is non-recrystallized fiber structure between transition layer and center layer, see Fig. 3. As shown in Fig. 3(a), the banded structure of the hot rolled plate without adding rare earth is concentrated in the center, the thickness is about 0.75 mm and is close to the 1/3 of the hot rolled plate. Figure 3(b) shows the banded structure is separated to several zones, central zones are grains, and the thickest of the banded structure is 0.24 mm.

3.1.3. Microstructure of the Finished Product

Figure 4 shows the microstructure of the finished product, the cold rolled plate has been recrystallized after annealing. As shown in Fig. 4(a), the microstructure of the finished product is disorganized without adding rare earth, the small grains are gathered around the large grains, the grain size is

| Furnace number | C     | Si    | Mn    | S     | AlS   | O     | N     | La   |
|---------------|-------|-------|-------|-------|-------|-------|-------|------|
| 1#            | 0.0024| 1.0   | 0.30  | 0.004 | 0.32  | 0.0013| 0.0029| –    |
| 2#            | 0.0025| 1.0   | 0.30  | 0.002 | 0.30  | 0.0011| 0.0030| 0.0020|

| Furnace number | $P_{1.556}/W\cdot kg^{-1}$ | $B_{50}/T$ |
|---------------|-----------------------------|-------------|
| 1#            | 4.214                       | 1.726       |
| 2#            | 3.814                       | 1.737       |

Table 1. Chemical composition of 50W600 (Wt%).

Table 2. Magnetic properties of the finished product.
obviously coarse after adding rare earth, with the number of the small grains decreases, the microstructure is more homogeneous, as shown in Fig. 4(b). The average grain size of the finished product is 44.23 \( \mu m \) without adding rare earth, while the average grain size is 58.47 \( \mu m \) with adding rare earth.

Figure 5 shows the orientation distribution function (ODF) section at constant \( \phi_2 = 45^\circ \) of the finished product, a texture characterized by strong \( \gamma \)-fiber texture and medium \( \alpha \)-fiber texture was produced after annealing. For \( \gamma \)-fiber texture, the intensity of 1# and 2# are 8.0 and 5.8 respectively. Quantitative analysis of texture is shown in Table 3, the favorable texture of (100) and (110) is increased by 3.57\%, unfavorable (111) texture is decreased by 4.16\% after adding rare earth.

3.2. Effect of Rare Earth Treatment on Inclusions

Figure 6 shows the inclusions of the hot rolled plate. Inclusions are mainly complex inclusions of AlN and sulfide

| Furnace number | (100) | (110) | (111) |
|---------------|-------|-------|-------|
| 1#            | 6.56  | 6.10  | 22.14 |
| 2#            | 8.94  | 7.29  | 17.98 |

Table 3. Texture content (%) of the finished product.
without adding rare earth. After the addition of rare earth, the composite inclusions of rare earth sulfide were found. Transmission electron microscopy is used for analyzing the fine inclusions of hot rolled plate with adding rare earth. Figure 7 presents the inclusion morphology of adding rare earth, we can see the precipitations of AlN in a single state, which are mainly lump in Fig. 7(a). The Energy spectrum selected area electron diffraction (SAED) pattern of the inclusions in Fig. 7(a) is indexed in Fig. 7(b), the lattice constant $a$ and $c$ of the inclusion are about 0.3 and 0.5 respectively. Therefore, the inclusion can be determined to be AlN (lattice constant of $a$ and $c$ are 0.3111 and 0.49792 respectively). Also, the complex precipitates of nitride and rare earth oxides were found in Fig. 7(c), which are mainly spherical. The rare earth oxides is the precipitate center of AlN, making AlN spheroidized and changing the morphology of the precipitates. Table 4 shows the inclusions distribution with adding rare earth and the others are without rare earth, the average size of the inclusions is increased marginally with adding rare earth, while the volume fraction and distribution density are decreased obviously, the total volume of the inclusion is reduced.

3.3. Discussion

It is obvious that the sulfur content of the steel is reduced from 40 ppm to 20 ppm and the effect of desulfurization is evident after adding rare earth. The sulfur content is decreased with the purity of molten steel increased, which leads to substantially reduce the number of the sulfide inclusions and the amount of inclusions in steel. The results have
The slab macrostructure is shown in Fig. 1(a), the columnar crystals are strong, while the equiaxed crystals are weak due to electromagnetic stirring cannot be proceeded, meanwhile the cooling and casting speeds of CSP are fast. Non-oriented electrical steel is high purity steel, due to silicon and aluminum are strong deoxidizing elements, and the oxygen activity in molten steel is extremely low, therefore the high melting point compounds such as RE₂O₂S and REₓSy will be preferentially precipitated with adding rare earth. Because the lattice misfit of rare earth oxides and sulfides with δ-Fe is the smallest, the nucleation undercooling is the smallest and much less than Al₂O₃, SiO₂ etc., it would be separated out before solidification of molten steel, and as the non-spontaneous crystal nucleus in the steel, it will expand the equiaxed crystal region and limit the development of the columnar crystals.²⁰,²¹ Therefore, solidification structure is improved by adding rare earth, as shown in Fig. 1(b).

The hot rolling reduction of CSP is much less than that of the conventional process, which causes the columnar crystals can not to be broken completely during the hot rolled process. The wide banded structure appears near the center of the hot rolled plate in Fig. 3(a), it is difficult to recrystallize during the annealing process, and the finished products have corrugated defect.²²,²³ Moreover, the energy consumed during the recovery process is increased with the severity of the band structure, the energy used to promote grain growth will be less. Therefore, the grain size of the finished product is smaller under the same annealing process. After adding rare earth, the development of the columnar crystals are suppressed, refined and broken, the banded structure of the hot rolled plate is decreased due to the structure heredity, and the width of banded structure is decreased from 0.75 mm to 0.24 mm, the annealing process is more prone to recrystallization and promote the grain growth in Fig. 3(b). Grain size affects the iron loss directly, because the smaller the grain is, the more the grain boundary is, the lattice distortion, the dislocation and the innerstress at the grain boundary are increased. The domain wall needs more energy at the grain boundary to move during the magnetization process, so the
hysteresis loss is large. With the growth of the grains, the number of the grain boundary is decreased, the resistance to the movement of the domain wall and the hysteresis loss is decreased, thus the iron loss is reduced by 0.4 W/kg after adding rare earth.

The inclusions shape have great influence on the magnetic properties, needle inclusions (such as long strips of MnS) or corrugated inclusions (such as AlN, TiN and other nitrides) have more influences on coercivity and hysteresis loss than spherical inclusions. Fig. 7 shows the lump AlN is separated out individually, while some inclusions of AlN are complex precipitates with rare earth oxides, which are spheroidal precipitates. The rare earth oxides is the precipitate center of AlN, adsorbing tiny AlN inclusions and then precipitates. The rare earth oxides is the precipitate center of AlN, adsorbing tiny AlN inclusions and then precipitates. The rare earth oxides is the precipitate center of AlN, adsorbing tiny AlN inclusions and then precipitates. The rare earth oxides is the precipitate center of AlN, adsorbing tiny AlN inclusions and then precipitates. The rare earth oxides is the precipitate center of AlN, adsorbing tiny AlN inclusions and then precipitates.

The study found[19] that the effect of the inclusion with size over 0.50 μm and under 0.05 μm is much smaller than that with the size from 0.05 μm to 0.50 μm, the number of the inclusions that prevent grain growth and pin magnetic domains are harmful to the non-metallic inclusions in steel. Guo Yanyong[19] studies that the inclusions size affecting the magnetic properties of electrical steel sheet is mainly between 0.11 μm with 0.4 μm, the size range is close to the thickness of the domain wall (usually a few hundred nm). These inclusions will pin domain wall during the magnetization process, that will prevent the rotation and motion of domain wall, therefore extra electrical energy is required to overcome this resistance, resulting in increasing the iron loss of electrical steel sheets. In this work, the average size of inclusions is about 1.4–1.5 μm, which has avoided the dangerous area, so it is the guarantee of improving the magnetic properties of electrical steel.

The unfavorable grains of (111) preferentially nucleate and grow near the inclusions and grain boundaries during the annealing process. The average grain size is increased with adding rare earth, the total length of the grain boundary is decreased as well as the inclusions distribution density and the total amount of inclusions are reduced. These can inhibit the nucleation and growth of the unfavorable grains of (111) effectively, the magnetic induction intensity and the favorable texture components are increased. So the magnetic induction intensity is increased by 0.011 T, see Table 2.

In conclusion, the coarse columnar crystals are inhibited and refined after adding element La, the banded structure of the hot rolled plate is reduced, therefore the average grain size of finished product is increased, meanwhile, the density and amount of inclusions are greatly reduced, and the AlN precipitate morphology was modified, which are the main reasons for improving magnetic properties.

4. Conclusions

The non-oriented electrical steel produced by CSP process was carried on rare earth treatment. As a result, the macrostructure of casting blank was refined and the banded structure of the hot rolled plate was reduced, sulfur content in the steel and the amount of sulfide precipitation were reduced as well as the microstructure of the finished product and the magnetic induction intensity were improved. The mechanisms are as follows:

(1) For slab macrostructure, the development of the columnar crystals is suppressed, refined and broken after adding rare earth element La, the equiaxed crystal ratio is increased, and the banded structure of the hot rolled plate is decreased.

(2) For the inclusions of the hot rolled plate, the density and amount of inclusions are greatly reduced, the precipitation morphology of AlN transformed from lump to spherical.

(3) For the microstructure of the finished product, the average grain size is increased by 14.24 μm with the addition of rare earth element La, meanwhile, the content of the favorable texture of (100) and (110) is increased, the unfavorable texture of (111) is reduced, and the magnetic properties are improved.

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