Effects of yttrium on the microstructure, texture, and magnetic properties of non-oriented 6.5 wt% Si steel sheets by a rolling process

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Abstract
Non-oriented 6.5 wt% Si steel thin sheets with three different yttrium (Y) contents (0, 0.012, and 0.03 wt%) were prepared by hot rolling, warm rolling, intermediate annealing, cold rolling and final annealing processes. The effects of the Y content on the microstructure, texture, and magnetic properties of cold-rolled 6.5 wt% Si steel sheets were studied by optical microscopy, scanning electron microscopy, energy-dispersive x-ray spectroscopy, and electron backscattered diffraction. The results showed that the sample with 0.012 wt% Y had the lowest volume fraction of inclusions, and Y played a role in purifying steel. The final average grain size of sheets decreased upon increasing the Y content. As the Y content increased, the \{100\} texture continuously weakened, and the overall intensity of the \eta(\langle100\rangle)\text{ //RD} texture increased first and then decreased, while the intensity of the detrimental \gamma(\langle111\rangle)\text{ //ND} texture decreased first and then increased. Adding an appropriate amount of Y optimized the recrystallization texture by promoting the occurrence of shear bands, which provided more nucleation sites for \eta-\text{fiber oriented grains}. When the Y content was 0.012 wt%, the magnetic induction \(B_{\text{50}}\) reached the maximum (1.64655 T) due to the enhanced \eta texture and weakened \gamma texture. The sample with 0.012 wt% Y showed the lowest core loss at high frequencies (>5 kHz) because of the favorable grain size. The addition of excess Y increased the number of inclusions and increased \gamma-\text{fiber oriented grain nucleation}, which deteriorated the magnetic properties of non-oriented 6.5 wt%Si steel.

1. Introduction

6.5 wt% Si steel has good soft magnetic properties, low core loss, high permeability, and near-zero magnetostriction, making it a preferred soft magnetic material for electromotors, transformers, and generators at high frequencies [1]; however, the formation of brittle B2 and DO3 ordered structures at room temperature restricts its industrial production and application [2, 3]. Rolling has the advantages of low cost, large output, controllable texture, stable performance, and low environmental pollution. The rolling preparation of 6.5 wt% Si steel sheets involves three stages: weakening its intrinsic brittleness, continuous improvement of the rolling and annealing method to ensure forming stability and texture optimization [4–7]. Although great progress has been made in the strip rolling of 6.5 wt% Si steel, its toughness, ductility and magnetic properties still need to be improved. On the one hand, the researchers improve the rolling and annealing process to promote and increase the disorder structures. On the other hand, they increase the plastic deformation ability of 6.5 wt% Si steel by adding small amounts of elements such as Ni, Mn, B, Nb, Cr, and rare earths (REs) [8–10].

The factors influencing the magnetic properties of electrical steels include the chemical composition, inclusions, grain size, and crystallographic texture [11, 12]. Generally, fine inclusions with a size of 30–100 nm have the strongest pinning effect on magnetic domains and grain boundaries, and they are unwanted detrimental residues in the final product [13]. In silicon steels, REs can be used as purifying agents and inclusion
modifiers, because they have strong affinities for oxygen and sulfur to form RE oxides, sulfides and oxysulfides, allowing them to be used as desulfurization and deoxidation agents [14]. The combined addition of RE elements and Al to non-oriented electrical steel decreased the volume fraction of fine inclusions and increased their size, which increased the grain size during annealing and reduced the iron loss [15]. When the Ce content in non-oriented 1.15 wt% Si steel was 0.003 wt%, the \{110\} \{001\} (Goss) texture became the strongest, magnetic induction was the highest, and the iron loss was the lowest [16]. In 6.47 wt% Si high silicon steel strips, when the Ce content was 0.017 wt%, the strong cube \((001)/(100)\), \{001\} \{120\}–\{001\} \{130\}, and Goss textures were obtained, and the \(\{111\}\//\text{ND}\) texture almost disappeared. Due to the purification effect of Ce, the grain size of the final sheets increased, and the magnetic properties were improved [17]. In 1.5 wt% Si non-oriented electrical steels, upon increasing La content, the final grain size and the magnetic induction first increased and then decreased, while the iron loss decreased. When the La content was 0.0055–0.0066 wt%, the cube texture intensity and magnetic induction were the highest [18, 19].

So far, the use of REs to improve the magnetic properties of non-oriented silicon steels has been limited to light REs (La or Ce), while the heavy RE yttrium (Y) has rarely been used. In this work, the effects of Y on the inclusions, microstructures, textures and magnetic properties of cold-rolled 6.5 wt% Si steel sheets were studied. The results provide a reference for the application of Y and developing non-oriented high-silicon steel with improved magnetic properties.

2. Experimental materials and methods

Three kinds of 6.5 wt% Si steel ingot with different Y contents were produced by a vacuum induction melting furnace, and the alloy composition was detected by inductively coupled plasma-atomic emission spectrometry (ICP-AES) as shown in table 1. The ingots were forged into 20 mm-thick plates at 1060 °C–900 °C, hot-rolled to 2.1 mm-thick bands at 1100 °C–850 °C, normalized at 900 °C for 3 min and quenched in oil. They were rolled to 0.6 mm-thick sheets at 500 °C. Afterward, the warm-rolled sheets were annealed at 950 °C for 10 min, quenched in oil, and then cold-rolled to 0.2 mm-thick sheets at 150 °C–200 °C. Finally, the sheets were annealed at 1100 °C for 6 min under a pure H₂ atmosphere. The final sheets were tailored to rectangular samples (300 mm length and 30 mm width) for magnetic testing.

The magnetic properties were measured using an electrical steel tester MPG200D, including the magnetic induction \(B_{50}\) (magnetic induction at 5000 A m\(^{-1}\)) and typical core losses at frequencies ranging from 40 Hz to 20 kHz. The core losses were \(P_{1/1000}, P_{1/10k}, P_{2/50k}, P_{2/50k}, P_{1/10k}, P_{0.5/20k}\). The metallographic structures were observed by an XLG-04 optical microscope. Inclusions were observed by a SIGMA Zeiss scanning electron microscope(SEM) and energy-dispersive spectroscopy (EDS) for elemental analysis. Fifty random field images were magnified by 2000 times and used to count the inclusion density distribution for each sample. The textures were analyzed by electron backscatter diffraction (EBSD; Oxford Instrument HKL Channel 5).

3. Results and discussion

3.1. Effect of Y on inclusions in the final sheets

Figure 1 presents the inclusion size distribution in the three final annealed samples with different Y contents. The statistical results indicate that the fine inclusion size distribution was mainly concentrated in the range 0–1.0 μm. Most inclusions in the sample without Y had sizes within the range of 0–0.5 μm, and the samples with Y had higher inclusion densities with sizes larger than 1.0 μm. The sample containing 0.012 wt% Y had the lowest inclusion density in the size range of 0.5–1.0 μm, and those in the size range of 0–0.5 μm were much lower than those in the sample without Y. By comparison, the size of inclusions in the sample without Y was distributed mostly in the range of 0.2–0.5 μm, while the inclusion distribution in the sample containing 0.03 wt% Y had a maximum value in the range of 0.5–1.0 μm. The addition of a moderate amount of Y purified 6.5 wt% Si steel and reduced the number of fine inclusions smaller than 0.5 μm in size; however, as the Y content further increased, the total number of inclusions increased.

| Sample | Si (wt%) | Y (wt%) | C | S | O | N | Fe |
|--------|---------|--------|---|---|---|---|----|
| 1      | 6.5     | 0      | 0.0043 | 0.0034 | 0.0034 | 0.0021 | Bal. |
| 2      | 6.5     | 0.012  | 0.0041 | 0.0029 | 0.0030 | 0.0020 | Bal. |
| 3      | 6.5     | 0.030  | 0.0040 | 0.0026 | 0.0029 | 0.0022 | Bal. |
Upon increasing the Y content, the average size of all oriented grains decreased. In the sample without Y, the main inclusions observed were TiN, Al2O3, and composite inclusions, and most inclusions were smaller than 0.2 \( \mu \text{m} \). In the sample with 0.012 wt% Y, the main inclusions observed were Y2O2S and composite inclusions of Y2O2S with Al2O3, and most inclusions had sizes in the range of 0.2–1.0 \( \mu \text{m} \). In the sample with 0.03 wt% Y, the main inclusions were Y2O2S and Y2O3, and the major inclusion size was larger than 1.0 \( \mu \text{m} \). Since Y has strong affinities for oxygen and sulfur to form Y oxide, sulfide and oxysulfide, the addition of Y reduced the number of fine inclusions such as Al2O3 and MnS, and increased the number of coarse composite inclusions in 6.5 wt% Si steel. However, as the Y content further increased, the total number of inclusions increased. Due to the fact that the atomic radius of Y (1.81 Å) is larger than that of Fe (1.24 Å), the solid solubility of Y is extremely low, and Y element has a strong tendency for grain boundary segregation. More inclusions containing Y formed and concentrated at the grain boundaries, which impeded grain boundary migration and hindered grain growth.

### 3.2. Effect of Y on the grain size of final sheets

Figure 3 presents the microstructure and grain size distribution of final sheets with different Y contents. For statistical analysis, eight pieces were taken from different areas of each sample and stacked together to observe the longitudinal sections. The average grain sizes of three samples with different Y contents were 194, 125, and 84 \( \mu \text{m} \), respectively. The average grain size decreased upon increasing the Y content, which indicates that Y addition played a grain refinement role in the final annealed 6.5%Si steel sheets.

EBSD analysis showed that the distribution and average size of \{100\}, \{110\}, and \{111\} oriented grains in the final annealed 6.5 wt%Si steel sheets containing different Y contents were obtained, as shown in figure 4. Upon increasing the Y content, the average size of all oriented grains decreased. In the sample without Y, the average size of the \{110\} grains was much larger than grains with other orientations. In the sample with 0.012 wt% Y, the \{100\} grains had the largest average size, and the \{111\} grains had the smallest grain size. In the sample with 0.03 wt% Y, the number of \{111\} grains increased, and their average grain size was the smallest. The \{110\} grains still had the largest average grain size but their number was small. Previous studies have shown that \{110\} atomic plane has the lowest surface energy under a pure hydrogen atmosphere. The \{110\} grains grew abnormally due to their low surface energy, so the grain size was much larger than the \{100\} and \{111\} grains. As for the reason why the \{100\} grain was larger than the \{111\} grain, the reduction rates of warm and cold rolling were 75% and 60%, respectively, which are both moderate reduction rates. The deformation energy storage was low, and the \{111\} recrystallization texture was suppressed. When the Y content was 0.03 wt%, the number of inclusions increased, and obvious grain refinement occurred. More grain boundaries offered more nucleation sites for \{111\} grains, so the number of \{111\} grains greatly increased.

### 3.3. Effect of Y on the texture of final sheets

Figure 5 shows the inverse pole figure (IPF) maps, \( \phi_2 = 45^\circ \) and \( \phi_2 = 0^\circ \) sections of the orientation distribution function (ODF) diagram of final sheets with different Y contents. The ODF diagram shows that a strong \( \lambda((001)/ ND) \) fiber texture existed in the sample without Y, which mainly includes \{001\} (610) and cube texture.
components. In addition, there were \{223\} (110), \{223\} (362), and \{332\} (113) components that were close to the \(\gamma\) (\{111\}//ND) fiber texture, as well as \{110\} (114) components that deviated 19° from the \{110\} (001) (Goss) orientation. In the sample containing 0.012 wt% Y, there was a strong cube texture and also a strong

Figure 2. Morphology and EDS analysis of typical inclusions in final annealed samples with different Y contents. (a), (b) without Y; (c), (d) 0.012 wt% Y; (e), (f) 0.03 wt% Y.
η ((100) //RD) fiber texture, which mainly included {610} (011) and Goss components, and the γ texture was greatly weakened. When the Y content increased to 0.03 wt%, the γ texture intensity increased obviously, while λ and η fiber textures were weakened.

Figure 6 shows the orientation densities of γ, λ, and η fibers of final sheets containing different Y contents. The intensity of the γ texture decreased first and then increased, and the intensity of {111}〈110〉 changed

Figure 3. Microstructure and grain size distribution of final sheets with different Y contents: (a) without Y; (b) 0.012 wt% Y; (c) 0.03 wt% Y; (d) grain size distribution.

Figure 4. Distribution and average sizes of {100}, {110}, and {111} oriented grains in the final annealed 6.5 wt% Si steel sheets with different Y contents: (a) without Y, (b) 0.012 wt% Y, (c) 0.03 wt% Y, (d) average sizes of special oriented grains.
significantly compared with that of \{111\}\{112\} as the Y content increased. The intensity of the \(\lambda\) fiber texture decreased gradually upon increasing the increase of Y content, especially the cube texture component. As for the \(\eta\) fiber texture, the intensity of the \{210\}\{001\}–\{110\}\{001\} texture components in the sample containing 0.012 wt% Y was obviously improved compared with that of the sample without Y. The overall intensity of the \(\eta\) fiber texture in the sample containing 0.03 wt% Y was the lowest. In summary, the \(\lambda\) fiber texture intensity decreased, and the \(\eta\) fiber texture intensity increased first and then decreased, whereas the \(\gamma\) fiber texture intensity decreased first and then increased upon increasing the Y content.

To study the causes of texture differences in the final sheets with different Y contents, the partially recrystallized cold-rolled sheets after annealing at 800 °C for 60 s were analyzed. Figure 5 shows the metallographic structures of partially recrystallized cold-rolled sheets, which were mainly composed of deformed microstructures. Shear bands are clearly visible in all samples, which are inclined at specific angles of 20–35° to the sheet surface and marked by red arrows in figure 7. By comparison, the density of shear bands greatly increased after Y addition. In the sample containing 0.012 wt% Y, the shear bands became larger and more concentrated, whereas the shear bands became smaller and more dispersed in the sample containing 0.03 wt% Y. The shear bands formed due to plastic instability caused by geometric softening, and their occurrence depended on several factors, including the grain size, orientation, solute, deformation temperature, and particles in the alloys [21–23]. Generally, large grains are favorable for the formation of shear bands, and the shear bands were more frequently observed in grains with a high Taylor factor, including grains with \{110\}\{110\} and \{111\}\{uvw\} orientations [24]. Most Y exists as compounds such as Y\(_2\)O\(_3\), YS, and Y\(_2\)O\(_2\)S in the Fe–6.5Si–0.03Y alloy [25]. These small non-deformable particles hindered dislocation movements, and the initially high work hardening rate of Fe–6.5 wt% Si steel containing Y was not maintained at high strains; therefore, the steel was prone to the occurrence of shear bands. However, when the Y content was 0.03 wt%, the
addition of excess Y led to grain refinement, and the smaller grain size was unfavorable for shear band formation. Increasing the number of shear bands can destroy \{111\} grains, inhibit the formation of \(\gamma\)-fiber texture components resulting from homogeneous deformation, and also contribute to the formation of favorable Goss and cube texture components in silicon steels \cite{24}; therefore, the density, size, and distribution of shear bands directly affected the development of the recrystallization texture during final annealing.

Figure 8 shows the IPF maps, \{001\} pole figures, and ODF diagrams (\(\varphi_2 = 45^\circ\) and \(\varphi_2 = 0^\circ\)) of partially recrystallized cold-rolled sheets with different Y contents. All samples had strong \(\gamma\)-fiber deformed textures, which increased with the Y content. At the same time, the recrystallization nucleation rates of 6.5 wt% Si steel sheets increased with the Y addition. As shown in figures 8(a) and (b), nucleation preferential occurred within shear bands, and the \(\eta\) and \(\lambda\) fiber oriented grains nucleated at shear bands in the \(\gamma\) deformation matrix (encircled by a red ellipse), and a few of them formed between the \{111\} and \{100\} deformed grains (encircled by a black ellipse). The deformation matrix with different orientations had different stored energy \(E\), and \(E_{\{111\}} > E_{\{100\}}\) \cite{26}. Moreover, the shear bands in the \(\gamma\)-fiber oriented grains with high deformation energy storage were preferential nucleation sites for \(\eta\)-fiber and \(\lambda\)-fiber oriented grains \cite{27-29}. At the grain boundary between \{111\} and \{100\} deformed grains, only a small number of grains (marked by black arrows) formed due to the lower energy storage. In the sample containing 0.012 wt% Y, a larger number of \(\eta\)-fiber oriented grains, including Goss and \{610\} \{001\}, nucleated at shear bands within \(\gamma\) deformation matrix, as shown in figure 8(d). According to previous studies, the nucleus with Goss orientation existed at the centers of sharp lattice curvature in shear bands, and the relationship between the Goss orientation and \{111\} \{112\} orientation of the deformed matrix was rotated 35° around the \{110\} axis \cite{30, 31}. Since the orientation within shear bands exhibited a strong dependence on the matrix orientation, there was a deviation between the orientation of the deformed matrix and \{111\} \{112\}. This caused the orientation of nuclei within shear bands to deviate from the Goss orientation, and then a strong \{610\} \{001\} texture component formed. According to the theory of oriented nucleation and oriented growth, special oriented grains with a high nucleation rate within the shear bands have a specific orientation relationship with the surrounding deformed matrix. They have high boundary mobility and grow rapidly, and ultimately determine the recrystallization texture \cite{24, 32}; therefore, the strongest \(\eta\)-fiber recrystallization texture formed in the sample containing 0.012 wt% Y.

For the sample containing 0.03 wt% Y as shown in figure 8(c), the formation of \(\eta\)- and \(\lambda\)-fiber oriented grains decreased because the addition of excess Y led to grain refinement. At the same time, the increasing number of inclusions also promoted \(\gamma\)-fiber oriented grain nucleation. As shown in figure 8(e), in the preferential nucleation area, \(\gamma\)-fiber oriented nuclei were dominant, and only a small number of \{001\} \{320\} nuclei formed. The recrystallization texture development was determined by nucleation and grain growth due to grain boundary migration related to RE inclusions and segregation \cite{33}. More RE inclusions and grain boundaries provided more preferential nucleation sites for \(\gamma\)-fiber oriented grains, and \(\gamma\) grains had a larger driving force when they grew into \(\gamma\) deformed grains with higher strain stored energy, and the strongest \(\gamma\)-fiber recrystallization texture finally formed in the sample containing 0.03 wt% Y.
3.4. Effect of Y on the magnetic properties of final sheets

Figure 9 shows the magnetic properties of the final annealed high silicon steel sheets containing different Y contents. As the Y content increased, the magnetic induction $B_{50}$ increased first and then decreased, as shown in figure 9(a). When the Y content was 0.012 wt%, the magnetic induction $B_{50}$ reached the highest value (1.64655 T). The crystallographic texture is an important factor that determines magnetic induction, so the final sheet containing 0.012 wt% Y had the highest $B_{50}$ value because it had the strongest $\eta$-fiber texture and also the weakest $\gamma$-fiber texture. In contrast, the final sheet containing 0.03 wt% Y had the lowest $B_{50}$ value because it had the strongest $\gamma$-fiber texture and the weakest $\lambda$ and $\eta$-fiber textures.

Figure 9(b) shows the core losses of the final sheets with different Y contents at different frequencies. The $P_{1.0}$ value gradually increased upon increasing the Y content at frequencies ranging from 50 to 1000 Hz. More shear bands increased the nucleation rate during annealing, which decreased the final grain size \[34\]. Then, more grain boundaries hindered domain movement. Y oxide and oxysulfide inclusions pinned magnetic domain wall movement, which increased the hysteresis loss and deteriorated the core loss; however, in the high-frequency field (>5 kHz), the core loss value first decreased and then increased upon increasing the Y content. The eddy current loss gradually dominated the core loss at higher frequencies, and the finer grain size helped reduce the eddy current loss; therefore, the sample containing 0.012 wt% Y had the lowest core losses because of its favorable grain size. Figure 9(c) shows the coercivities of the final sheets with different Y contents at different frequencies.

Figure 8. IPF maps (orientation deviation angle $\leq 15^\circ$), [001] pole figures and ODFs ($\varphi_2 = 45^\circ$ and $\varphi_2 = 0^\circ$) of partially recrystallized cold-rolled sheets (a) without Y; (b) 0.012 wt% Y; (c) 0.03 wt% Y; (d) magnified map, PF, and ODFs of local region A in (b); (e) magnified map, PF, and ODFs of local region B in (c).
frequencies and the change trend is almost consistent with the core loss. This is because the finer grain size results in a higher coercivity [35]. The sample containing 0.03 wt% Y had the highest core losses at all frequencies because it had the highest inclusion density and strongest harmful $\gamma$-fiber texture. This indicates that the addition of excessive Y deteriorated the magnetic properties of 6.5 wt% Si steel sheets.

4. Conclusions

(1) The moderate addition of Y purified 6.5 wt% Si steel and reduced the number of fine inclusions; however, as the Y content further increased, the total number of inclusions increased. The average grain size of the final sheets gradually decreased upon increasing the Y content, indicating that Y had a grain refining effect.

(2) As the Y content increased, the $\{100\}$ texture of the final sheets was continuously weakened, and the overall intensity of the $\eta$ texture increased first and then decreased, while the intensity of harmful $\gamma$ decreased first and then increased. Adding an appropriate amount of Y optimized the recrystallization texture by promoting the formation of shear bands, which provided more nucleation sites for $\eta$-fiber oriented grains.

(3) When the Y content was 0.012 wt%, the magnetic induction $B_{50}$ reached the maximum (1.64655 T) due to the enhanced $\eta$ texture and weakened $\gamma$ texture. The sample with 0.012 wt% Y showed the lowest core losses at high frequencies (>5 kHz) because of its favorable grain size. The addition of excessive Y increased the number of inclusions and also increased the $\gamma$-fiber oriented grain nucleation, which deteriorated the magnetic properties of non-oriented 6.5 wt% Si steel.

Acknowledgments

This work was supported by the National Natural Science Foundation of China (No. 51704131).
Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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