Effect of {114}<418> Texture on Abnormal Grain Growth during Secondary Recrystallization in Grain-Oriented Silicon Steel Postprint

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Abstract

EBSD technology was utilized to investigate the secondary recrystallization behavior of thin-gauge HiB steel samples under high-reduction cold rolling conditions, thereby elucidating the reasons for the difficulty in secondary recrystallization occurrence in thin-gauge specimens. Inhibitors were introduced through nitriding treatment to facilitate smooth secondary recrystallization, and the influence of nitriding amount on secondary recrystallization in thin-gauge samples, as well as the effect of {114}<418> texture on abnormal grain growth of brass-oriented and Goss-oriented grains, were examined. The results demonstrate that during secondary recrystallization annealing, grains near the {114}<418> orientation in the surface layers on both sides exhibit a pronounced growth advantage compared to grains with other orientations, and propagate toward the center layer. If the inhibitor strength in the sample surface layer is insufficient, these grains can readily grow to the center layer of the sample and engulf the secondary recrystallization nuclei, which is detrimental to secondary recrystallization. Supplementing inhibitors through nitriding can effectively suppress the growth of surface grains, thereby promoting secondary recrystallization and enhancing magnetic properties. In the initial stage of secondary recrystallization, grains near the {114}<418> orientation tend to persist at the centers of abnormally growing tilted Goss-oriented and brass-oriented grains, forming island grains that hinder their abnormal growth; however, the influence of grains of this orientation on the abnormal growth of Goss-oriented grains is relatively minor.

Full Text

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Abstract

To investigate the poor secondary recrystallization behavior in thin-gauged grain-oriented steel, EBSD technique was applied to reveal the grain growth behavior of thin-gauged samples processed by HiB steel method under high cold-rolling reduction. Nitriding treatments with different durations were conducted to ensure the occurrence of secondary recrystallization in thin-gauged samples and to determine the effect of nitrogen content, with particular attention paid to the influence of {114}<418> texture on the abnormal growth of Brass and Goss grains. Results show that during secondary recrystallization annealing, grains near the {114}<418> orientation in the surface layers exhibit significant growth advantage over other oriented grains and grow toward the center layer. If the inhibitor strength in the surface layer is insufficient, these grains can readily grow to the center layer and swallow the nuclei for secondary recrystallization, thereby preventing secondary recrystallization. Supplementing inhibitors through nitriding treatment effectively suppresses surface grain growth, thus promoting secondary recrystallization and improving magnetic properties. In the initial stage of secondary recrystallization, grains near the {114}<418> orientation tend to be retained as island grains within the centers of abnormally grown scattered Goss and Brass-oriented grains, which is detrimental to their abnormal growth. However, this orientation has a relatively weaker effect on the abnormal growth of Goss-oriented grains.

KEY WORDS {114}<418> texture; thin-gauged grain-oriented silicon steel; abnormal growth; inhibitor

1. Introduction

Grain-oriented silicon steel utilizes the secondary recrystallization principle to obtain sharp Goss texture, thereby achieving excellent magnetic properties. The microstructure and texture after primary recrystallization significantly influence secondary recrystallization behavior, with different types of grain-oriented silicon steel exhibiting distinct primary recrystallization texture characteristics. Conventional grain-oriented (CGO) steel develops cube texture and γ-fiber texture dominated by {111}<110> after two-stage moderate cold rolling, while traditional high-permeability grain-oriented (HiB) steel processed by single-stage cold rolling with 87% reduction produces strong 25° rotated cube texture and γ-fiber texture dominated by {111}<112>. For both low-temperature nitriding-type and thin-gauged grain-oriented silicon steels, which undergo cold rolling reductions above 90%, the primary recrystallization textures are dominated by {114}<418> and γ-fiber textures. The difference lies in that the γ-fiber texture weakens in low-temperature nitriding-type steel but strengthens significantly in thin-gauged steel. As grain-oriented silicon steel continues to develop toward low-temperature slab heating and thin-gauge directions, investigating the effect of {114}<418> texture on secondary recrystallization behavior holds important theoretical significance.

During thin-gauged grain-oriented silicon steel production, two critical issues arise. First, inhibitors in the sample surface layer mature too rapidly during secondary recrystallization annealing, making surface grains prone to excessive growth that subsequently swallows Goss-oriented nuclei in the subsurface layer, which is detrimental to secondary recrystallization. However, the textural characteristics of these growing surface grains have not been reported. Second, excessive cold rolling reduction decreases the number of Goss-oriented grains while increasing Brass-oriented grains. During secondary recrystallization annealing, competition between Brass and Goss-oriented grains for abnormal growth deteriorates magnetic properties. Therefore, the key to improving performance lies in suppressing abnormal growth of Brass-oriented or near-Brass-oriented grains during secondary recrystallization annealing. In Fe-3%Si (mass fraction) alloys subjected to high cold rolling reduction, strong {114}<418> recrystallization texture emerges. Similar to {111}<112>, {114}<418> also exhibits a $\Sigma 9$ grain boundary relationship with Goss orientation. While {111}<112> recrystallization texture is generally considered beneficial for abnormal growth of Goss-oriented grains, the influence of {114}<418> recrystallization texture on abnormal growth of both Goss and Brass-oriented grains remains unclear. Consequently, this work investigates secondary recrystallization behavior in thin-gauged grain-oriented silicon steel with and without inhibitor addition through interrupted annealing experiments, analyzing orientation relationships between abnormally grown Goss/Brass-oriented grains and their internal island grains to elucidate the effect of {114}<418>-oriented grains on abnormal growth. The findings provide theoretical guidance for enhancing Goss texture sharpness after secondary recrystallization in thin-gauged grain-oriented silicon steel.

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2. Experimental

The experimental material was hot-rolled sheet of grain-oriented silicon steel with MnS+AlN inhibitors. The main chemical composition (mass fraction, %) was: C 0.049-0.058, Si 3.00-3.33, Mn 0.06-0.10, P 0.011-0.019, S 0.015-0.026, Als 0.014-0.019, N 0.0055-0.0075, with Fe balance. The hot-rolled sheet (2.3 mm thick) was normalized and then cold-rolled in a single pass to 0.23 mm thickness with 90% reduction. After decarburization annealing at 880 °C for 5 min, one group of samples underwent nitriding treatment at 750 °C for 30, 60, and 90 s, while another group received no nitriding treatment. Both groups were finally coated with MgO and subjected to high-temperature annealing. During high-temperature annealing, samples were heated from room temperature to 950 °C at 30 °C/h in a N_2:H_2 = 1:3 atmosphere, then from 950 °C to 1150 °C at 10 °C/h. This study employed an "interruption method" to investigate microstructural and textural evolution during continuous heating, with samples withdrawn from the furnace every 25 °C starting from 950 °C. Grain structure and local orientations of decarburized and interrupted samples were examined using a LEO-1450 scanning electron microscope (SEM) equipped with a Channel 5 electron backscatter diffraction (EBSD) system. After completing high-temperature annealing, magnetic properties were measured using a NIM-2000E silicon steel sheet magnetic measurement instrument to determine B_8 (magnetic flux density at magnetic field strength of 800 A/m, in Tesla).

2.1 Textures of Cold-Rolled and Decarburized Sheets

Figure 1 [Figure 1: see original paper] shows EBSD orientation imaging maps of the cold-rolled sheet (maximum deviation angle of 15° for each orientation). As seen in Fig. 1a, a certain amount of {114}<418> component exists within {112}<110> deformed grains, indicating a transformation relationship between {114}<418> and {112}<110> orientations during deformation. Figure 1b displays the distribution of orientations from the EBSD map in a {100} pole figure. Figure 1c reveals that the cold-rolling texture consists of typical α-fiber and γ-fiber textures, with the γ-fiber texture strongest at {111}<110> and weaker at {111}<112>, which results from excessive rolling reduction. At this stage, the {114}<418> texture intensity remains relatively weak.

Figure 2 [Figure 2: see original paper] presents EBSD orientation imaging maps of the primary recrystallization sample after 90% cold rolling reduction (maximum deviation angle of 15° for each orientation). Unlike conventional CGO steel with cube texture or HiB steel with 25% reduction showing 25° rotated cube texture, strong {114}<418> texture emerges in the primary recrystallization texture, accompanied by significantly enhanced {111}<112> texture intensity. The large orientation difference between {111}<112> and

{111}<110> enables formation of highly mobile high-angle grain boundaries. The strong {111}<110> texture in the cold-rolled structure facilitates growth of {111}<112>-oriented nuclei into the {111}<110> deformed matrix during recrystallization. Therefore, the strong {111}<110> cold-rolling texture is responsible for the strong {111}<112> primary recrystallization texture. Excessive cold rolling reduction introduces small amounts of {114}<418> component in α-fiber-oriented deformed grains. Since deformed grains on the α-fiber orientation line possess low stored energy, their nucleation and growth during recrystallization proceed slowly. Moreover, {114}<418> grains readily form high-angle grain boundaries with α-fiber-oriented deformed grains, giving {114}<418>-oriented grains obvious growth advantages over α-fiber-oriented grains. This leads to accumulation of α-fiber texture toward the {114}<418> orientation in the primary recrystallization texture.

Figure 3 [Figure 3: see original paper] shows grain size distributions for {114}<418> and {111}<112> grains (maximum deviation angle of 15°) in the primary recrystallization microstructure. Calculations reveal average grain sizes of 20.2 m for {114}<418> grains and 15.4 m for {111}<112> grains, indicating that {114}<418> grains are slightly larger. Deformed grains on the γ-fiber possess high stored energy, resulting in numerous recrystallization nuclei. As shown in Fig. 2a, {111}<112> grains are primarily surrounded by {114}<418>, {111}<110>, and {111}<112> grains. Statistical analysis indicates that boundaries between {111}<112> grains and these neighboring grains are mainly low-angle boundaries, $\Sigma 3$ boundaries, and certain high-angle boundaries, all of which are difficult to migrate. Consequently, {111}<112> grains in the primary recrystallization microstructure remain relatively small. In contrast, {114}<418> grains nucleate and grow within deformed grains with low stored energy on the α-fiber orientation line, readily forming high-angle grain boundaries with α-fiber-oriented deformed grains for rapid growth. This results in significantly larger {114}<418> grain sizes after primary recrystallization.

2.2 Grain Orientation and Size During High-Temperature Annealing of Non-Nitrided Samples

Figure 4 [Figure 4: see original paper] shows EBSD orientation imaging maps of non-nitrided samples at different temperatures during high-temperature annealing. The results demonstrate that compared with grains in the center layer, grains in both surface layers exhibit obvious growth advantages, with the vast majority being orientations near {114}<418>. As temperature increases, surface layer grains gradually grow toward the center layer, progressively strengthening the {114}<418> texture. The more pronounced growth of surface grains compared to center grains occurs because inhibitor coarsening proceeds faster in the surface layer under H_2 atmosphere. The predominance of {114}<418>-oriented grains in the growing surface layer stems from their larger size in the primary recrystallization microstructure, which provides a growth advantage.

2.3 Effect of Nitriding Time on B_8 After High-Temperature Annealing

Figure 5 [Figure 5: see original paper] shows macrostructures of samples after secondary recrystallization under different nitriding times. Measurements indicate that B_8 increases with prolonged nitriding time. Without nitriding treatment before high-temperature annealing, secondary recrystallization fails to occur, yielding a B_8 of only 1.588 T with a nearly fully fine-grained macrostructure. After short-duration nitriding (30 s), secondary recrystallization proceeds successfully, with B_8 significantly increasing to 1.746 T. However, this value remains suboptimal, as the macrostructure shows numerous grains that are small but relatively uniform. Further extending nitriding time gradually increases B_8, reaching 1.846 T at 60 s and 1.900 T at 90 s, with the latter showing a macrostructure characterized by fewer, larger grains.

2.4 Characteristics of Island Grains Within Abnormally Grown Goss and Brass-Oriented Grains

Figure 6 [Figure 6: see original paper] presents EBSD orientation imaging maps of a sample nitrided for 60 s and heated to 1010 °C during high-temperature annealing. Figures 6a and 6b show the EBSD orientation map and {100} pole figure of an abnormally grown Goss-oriented grain, while Figs. 6c and 6d display those of an abnormally grown Brass-oriented grain. Island grains in Goss-oriented grains are predominantly distributed near the sample surface, whereas island grains in Brass-oriented grains appear not only near the surface but also abundantly in the center region. This work statistically analyzed 26 island grains within 8 abnormally grown Brass-oriented grains and 27 island grains within 11 abnormally grown Goss-oriented grains during the initial secondary recrystallization stage. The statistical results in Figs. 6e and 6f reveal that island grains in both Goss and Brass-oriented grains are predominantly {114}<418>-oriented.

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3. Analysis and Discussion

Regarding {114}<418> texture formation, it is generally accepted that strong α-fiber texture develops after heavy deformation, with small amounts of {114}<418> component forming within α-fiber deformed grains that serve as recrystallization nuclei. However, the origin of {114}<418> orientation remains inconclusive. Gobernado et al. observed {113}<631> orientation (approximating {114}<418>) within {100}<011> deformed grains in interstitial-free steel through cross-rolling and EBSD characterization, though significant

{113}<631> texture did not form after recrystallization. This study could not eliminate the influence of cube orientation, which may transform to rotated cube orientation during cold rolling. To avoid interference from other orientations, Toge et al. investigated texture evolution during warm rolling of Fe-3%Si alloy (100)[011] single crystals, demonstrating that dynamic strain aging during warm rolling of C-containing silicon steel creates 35° deformation or shear bands within rotated cube grains, ultimately forming strong {114} texture upon annealing. Homma et al. studied Fe-3%Si alloy (100)[011] and (112)[110] single crystals as well as polycrystals, finding that strong α-fiber texture forms under heavy deformation with {114}<418> orientation present internally, leading to strong {114}<418> texture after recrystallization. Since 230 mm thick continuous casting slabs of electrical steel contain numerous {100} columnar grains that develop cube and 25° rotated cube orientations after cold rolling with various reductions, the strong {114}<418> texture in HiB steel after heavy cold rolling and annealing may originate from either {100} columnar grains in the casting slab or special orientations within pure α-fiber deformed grains under heavy reduction.

In primary recrystallization microstructures of grain-oriented silicon steel, Goss texture intensity gradually weakens from surface to center layer, while scattered Goss texture intensity increases. Nucleation sites for secondary recrystallization grains occur at approximately 20% of the sheet thickness. Without nitriding treatment, secondary recrystallization fails because insufficient surface inhibitors and their premature coarsening allow {114}<418>-oriented surface grains to grow to the center layer, swallowing secondary recrystallization nuclei. Nitriding treatment suppresses surface grain growth during secondary recrystallization annealing, promoting secondary recrystallization. After short-duration nitriding (30 s), secondary recrystallization occurs with significantly increased B_8, though the value remains suboptimal due to insufficient surface inhibitors. While supplemented inhibitors restrain surface grain growth, surface grains still extend to the subsurface layer, consuming predominantly subsurface Goss-oriented nuclei and enabling abnormal growth of near-Brass-oriented grains in the center layer. Since near-Brass-oriented grains are more numerous than Goss-oriented grains, abnormal growth of center-layer near-Brass-oriented grains substantially increases the number of secondary recrystallization nuclei, resulting in a final macrostructure with numerous small but relatively uniform grains. Further increasing nitriding content more effectively suppresses surface grain growth, preventing surface grains from reaching the subsurface layer to consume Goss nuclei when secondary recrystallization occurs. Consequently, Goss-oriented grains can undergo abnormal growth. Due to excessive cold rolling reduction in thin-gauged samples reducing the number of Goss-oriented grains in the primary recrystallization texture, abnormal growth of Goss-oriented grains yields a final macrostructure with large grains and high B_8.

Island grains within abnormally grown grains form primarily for two reasons: large original island grain size and low-mobility grain boundaries between island grains and abnormally grown grains. Rajmohan and Szpunar found that island grains after secondary recrystallization are mostly surrounded by low-mobility grain boundaries with misorientation angles less than 20° or greater than 45° (grain boundaries marked with yellow lines in Figs. 6a and 6c). Figure 7 [Figure 7: see original paper] shows differences in misorientation angle distribution between Brass-oriented grains and {114}<418> island grains located at different positions within Brass-oriented grains. No grain boundaries with misorientation less than 20° form between Brass-oriented grains and {114}<418> grains. {114}<418> island grains in the center of Brass-oriented grains predominantly form high-angle boundaries with misorientation greater than 45°, whereas those at the sample surface form more boundaries with 20°-45° misorientation than >45° boundaries. As previously mentioned, surface {114}<418> grains exhibit obvious growth advantages over other orientations during secondary recrystallization annealing. Therefore, island grains at contact positions between Goss/Brass-oriented grains and the surface result from large surface {114}<418> grains that are difficult for Goss and Brass-oriented grains to consume, ultimately remaining within them as island grains. {114}<418> island grains in the center of Brass-oriented grains predominantly form >45° low-mobility boundaries, indicating their formation results from the tendency to create low-mobility boundaries with misorientation greater than 45° between Brass-oriented and {114}<418>-oriented grains. This work observed no {114}<418> island grains in the center of Goss-oriented grains but numerous {114}<418>-oriented island grains in the center of Brass-oriented grains, demonstrating that Goss-oriented grains possess stronger ability to consume {114}<418> grains than Brass-oriented grains. During thin-gauged grain-oriented silicon steel production, excessive cold rolling reduction increases Brass-oriented grains while decreasing Goss-oriented grains in the primary recrystallization texture, making it easier to form certain amounts of large Brass-oriented grains after secondary recrystallization, thereby deteriorating magnetic properties. Large cold rolling reduction also creates strong {114}<418> texture in the primary recrystallization texture. The stronger ability of Goss-oriented grains to consume {114}<418> grains compared to Brass-oriented grains may therefore be beneficial for improving Goss texture sharpness after secondary recrystallization.

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4. Conclusions

(1) Distinct from the cube texture in conventional CGO steel and the 25° rotated cube texture in HiB steel with 87% reduction, the Fe-3%Si alloy in this work develops strong {114}<418> texture after cold rolling and annealing with 90% heavy reduction. After primary recrystallization, {114}<418> grains are slightly larger than grains on the {111} orientation line.

(2) For the investigated composition during high-temperature annealing, insufficient surface inhibitors lead to premature coarsening, enabling {114}<418>-oriented grains to grow significantly based on their size

advantage until reaching the center layer and consuming {111} grains and secondary recrystallization nuclei, preventing secondary recrystallization.

(3) Nucleation sites for secondary recrystallization cores greatly influence final Goss texture sharpness during high-temperature annealing. Nucleation in the subsurface layer favors enhanced Goss texture sharpness, whereas nucleation in the center layer reduces it. Therefore, controlling excessive surface grain growth by strengthening surface inhibitors is crucial for improving final magnetic properties.

(4) Due to their relatively large size, {114}<418> grains are detrimental to abnormal growth of both Goss and Brass-oriented grains. However, Goss-oriented grains exhibit slightly stronger ability to consume {114}<418> grains than Brass-oriented grains, as evidenced by {114}<418>-oriented island grains being predominantly located at surface contact positions in Goss-oriented grains and within the interior of Brass-oriented grains. This occurs because grains near Brass orientation readily form low-mobility grain boundaries with misorientation greater than 45° with {114}<418>-oriented grains, thereby hindering abnormal growth of Brass-oriented grains.

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