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Abstract. 0.08mm ultra-thin silicon steel is prepared with 0.30mm commercial grain-oriented silicon steel as raw material, and the evolutions of η-fiber microstructure and texture are investigated with emphasis. During rolling deformation, initial Goss orientation shows crystal rotation to {111}<112> orientation. With the increase of rolling reduction, the retention of Goss orientation is reduced and more observed near the sheet surface. For initial deviated Goss oriented grains, {111}<112> and η-fiber oriented areas are also observed in 0.08mm rolled sheet, while the accuracy and concentration of these two components are decreased. During annealing, η-fiber nucleation superiority in γ-fiber deformed regions is significant, and thus contributes to strong η-fiber recrystallization texture. When the rolled sheets are heated at 800°C for 15 min, strong η-fiber recrystallization texture and uniform microstructure is beneficial for the magnetic properties of ultrathin silicon steel sheets.

1. Introduction

Ultra-thin silicon steel with the thickness lower than 0.10 mm is paid special attention for its applications in intermediate and high frequency devices [1-2]. It could be prepared by rolling and annealing grain-oriented silicon steel sheets with strong Goss (\{011\}<100>) texture[3-6]. The low iron losses under medium and high frequencies show the effect of ultra-low steel sheet thickness and are influenced by microstructure, and the magnetic induction could be improved by texture optimization, namely increasing the intensity of η-fiber (\{0kl\}<100>, <100>// rolling direction) recrystallization texture[3,7-9].

Considering the strong Goss texture and coarse microstructure of raw material, the deformation and recrystallization behaviors of Goss oriented grains greatly affect the microstructure as well as texture evolution. It is well known that Goss orientation rotate to \{111\}<112> orientation under rolling deformation, then Goss and other η-fiber nuclei could be observed inside shear bands in \{111\}<112> matrix during annealing [10-14]. Given that deformation behavior of ultra-thin silicon steel production is distinct from common polycrystals, in our earlier work, we discussed the deformed microstructure and orientation characteristics as well as recrystallization behaviors of initial Goss oriented grains in 0.075mm ultra-thin silicon steel [15].

In this study, ultra-thin silicon steel was prepared with 0.30mm commercial grain-oriented silicon steel, which is lower than that used in our earlier work. The aim is to clarify the orientation and
microstructure evolution by tracing the deformation process with the increasing rolling reduction, meanwhile the difference between the surface and interior of the rolled sheets was paid attention. It is worthy to note that rolling reduction is changed when the thickness of raw material is different, thus this work could expand the study of ultra-thin silicon steel production.

2. Experimental procedure
Plates of 30 mm (along transverse direction, TD) × 300 mm (along rolling direction, RD) were cut from a 0.30mm commercial grain-oriented silicon steel and were used as raw material to prepare ultra-thin silicon steel. The initial magnetic properties were shown to be $B_s=1.889T$ and $P_{17/50}=1.36 \text{ W/kg}$. $B_s$ means magnetic induction corresponding to 800A/m magnetic field intensity, and $P_{17/50}$ means core loss of sample per kg at maximum magnetic induction 1.7T and frequency 50Hz. After being moving coating layer, the starting material was 0.28mm thick. Cold rolling was performed by multiple passes to obtain 0.18mm, 0.1mm and 0.08mm rolled samples, corresponding to reductions of 35.7%, 64.3% and 71.4%. The 0.08mm rolled sheets were annealed at 700℃ and 800℃ to obtain partially and fully recrystallized microstructures, and the magnetic properties of fully recrystallized sheet were measured.

The microstructure and texture evolutions were evaluated with a Zeiss GeminiSEM500 field emission scanning electron microscope (SEM) equipped with EBSD system. The deformed samples were measured in rolling and lateral planes, and the measurements were performed in different stages of deformation. The annealed samples were measured in rolling plane. To decrease possible statistical error due to coarse initial grains in raw material, more than 5 specimens for each sample were evaluated, and representative results are shown in this paper. The tolerance angles for Goss and \{021\}<100> orientations are 10° to avoid overlap, and the angle for other orientations is 15°.

3. Results and discussions
The three image maps in Fig.1 represent the rolled sheets under reductions of 35.7%, 64.3% and 71.4% respectively. The crystal rotation trend from Goss to \{111\}<112> is clearly depicted, and the transiting orientation components between Goss and \{111\}<112> are displayed as black dots in the pole figures. With the increasing rolling reduction, \{111\}<112> oriented areas gradually occupy the majority of deformed microstructure, and the retention of Goss orientation is reduced. However, when the rolling reduction reaches 71.4%, Fig.1(c) still shows many Goss oriented areas, parts of which can be deduced to be retained from the initial Goss grain. Some Goss oriented areas are surrounded by high-angle (>15°) boundaries (black lines in orientation image maps) as small discrete regions, thus relating to high orientation gradient.
Fig. 1 Deformed microstructure and texture evolution of initial Goss grains in rolling planes. 35.7% rolling reduction: a) orientation image map; d) {200} and {111} pole figures; 64.3% rolling reduction: b) orientation image map; e) {200} and {111} pole figures; 71.4% rolling reduction: c) orientation image map; f) {200} and {111} pole figures.

Fig. 2 illustrates the deformation process in lateral plane, and the same gradual changes from initial Goss orientation to the end {111}<112> orientation are observed. In Fig. 2(b), the retained Goss oriented areas align in specific direction, which is similar to the distribution characteristic of shear band. The slip traces in the same direction are observed in Fig. 2(c), and they may be related to the activation of specific {112}<111> slip system. In Fig. 2(c), it is observed that Goss and minor {021}<100> areas are mainly located near the sheet surface.

Fig. 2 Deformed microstructure and texture evolution of initial Goss grains in lateral planes. 35.7% rolling reduction: a) orientation image map; d) {200} and {111} pole figures; 64.3% rolling reduction: b) orientation image map; e) {200} and {111} pole figures; 71.4% rolling reduction: c) orientation image map; f) {200} and {111} pole figures.
It is generally considered that shear bands usually occur in large γ-fiber deformed grains under the rolling reduction of 70%, and η-fiber oriented areas can be achieved inside shear bands. Shear band is not clearly identified in Fig.2(c), while its existence could not be denied for possible measurement error. However, those Goss and minor {021}<100> areas without extending trace into the interior of the rolled sheet are largely by the retention. Therefore, it can be inferred that the retention of initial Goss orientation is increased by the surface shear.

For the commercial grain-oriented silicon steel used as raw material, deviated Goss grains with the orientation deviating from exact Goss for 5°-15° degrees commonly exist. Typical deformed microstructures of this kind of deviated Goss grains are given in Fig.3. Similar orientation transition route towards {111}<112> is displayed. The majority of deformed microstructure also orients as {111}<112>, while more ‘white’ areas with transitional orientation are shown in Fig.3(a) and Fig.3(c). That corresponds to the lower fraction of {111}<112> deformed area and lower accuracy and concentration of {111}<112> deformation texture. Goss and {021}<100> oriented regions are observed in rolling plane. Compared with initial Goss grains in Fig.1 and Fig.2, the fraction of {021}<100> area is higher, meanwhile more η-fiber areas are located in the interior of rolled sheet. Given that no marked shear bands are observed, these η-fiber orientations are mostly linked to the orientation transition route of initial deviated Goss grain during rolling deformation. (001)<110> oriented deformation twin is depicted in Fig.2(b) in 2(d), and it is also observed in our earlier work [15].

Based on the producing method of ultra-thin silicon steel, the magnetic induction of final product is improved by strengthening η-fiber recrystallization texture. Therefore, we analyzed the fraction of η-fiber deformed areas in the regions of Fig.2(c) and Fig.3(b), and the distribution is shown in Fig.4. When Goss and deviated Goss oriented initial grains are deformed to the same strain, the area fraction of η-fiber (15° deviation permitted) region is close. However, the accuracy of η-fiber orientation is lower in the initial deviated Goss grain. Given that during annealing, nuclei originate from those pre-existed areas in deformed matrix, to choose the raw material with more exact Goss grains is more beneficial for increasing the intensity of accurate η-fiber texture.
Fig. 4. The distribution of η-fiber area fraction in different initial oriented grains.

Fig. 5. Recrystallized microstructures and micro-textures of 0.08mm silicon steel. Partially recrystallized sample: a) orientation image map; b) band contrast map; c) {200} and {111} pole figures; Fully recrystallized sample: d) orientation image map; e) ODFs at φ₂=0° and 45° sections.
After being annealed at 700°C, plenty of $\eta$-fiber nuclei in $\gamma$-fiber deformed matrix are achieved, as shown in Fig.5(a-c). New grains may be due to the retention or nucleation inside shear bands, and those locating at the boundaries in the left part of Fig.5(a-b) should be linked to the retaining of initial orientation [16]. The nucleation advantage of $\eta$-fiber grains leads to the strong $\eta$-fiber texture in fully recrystallized sample in Fig.5(d-e), which was heated at 800°C for 15min. The magnetic induction $B_8$ of this fully recrystallized sample is measured to be 1.802T. The microstructure is uniform, contributing to the iron loss $P_{15/400}$ of 12.6 W/kg, which is core loss of sample per kg at maximum magnetic induction 1.5T and frequency 400Hz. The average grain size is lower than 100 $\mu$m, and non-$\eta$-fiber grains do not show obvious growth advantage, so the iron loss is potential to be decreased by modifying the annealing parameters.

4. Conclusions
During rolling deformation, initial Goss orientation shows crystal rotations to \{111\}<112> orientation. With the increasing rolling reduction, the retention of Goss orientation is reduced and more observed near the sheet surface. For initial deviated Goss oriented grains, \{111\}<112> and $\eta$-fiber oriented areas are also observed in 0.08mm rolled sheet, while the accuracy and concentration of these two components are decreased, and more \{021\}<100> oriented areas are shown in the deformed sheet. After annealing, $\eta$-fiber nucleation superiority in $\gamma$-fiber deformed regions is significant, and thus contributes to strong $\eta$-fiber texture in the fully recrystallized sheet heated at 800°C for 15min. Non-$\eta$-fiber grains do not show obvious size advantage, strong $\eta$-fiber recrystallization texture and uniform microstructure is beneficial for the magnetic properties of ultrathin silicon steel sheets.

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