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## Secondary recrystallization behavior in a twin-roll cast grain-oriented electrical steel



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## ABSTRACT

The microstructure and texture evolution along the processing was investigated with a particular focus on the secondary recrystallization behavior in a 0.23 mm-thick twin-roll cast grain-oriented electrical steel. A striking feature is that Goss orientation originated during twin-roll casting as a result of shear deformation and it was further enhanced during hot rolling and normalizing. After primary recrystallization annealing, a homogeneous microstructure associated with a sharp y-fiber texture was produced. During secondary recrystallization annealing, the  $\gamma$ -fiber texture was first strengthened and weakened with increasing temperature prior to the onset of secondary recrystallization. Goss grains always exhibited more 20-45° misoriented boundaries than the matrix. The matrix was quite stable during secondary recrystallization with the aid of dense inhibitors. Finally, a complete secondary recrystallization microstructure consisting of large Goss grains was produced. The grain boundary characteristics distribution indicated that the high energy model was responsible for the abnormal growth of Goss grains under the present conditions.

#### 1. Introduction

The conventional processing of grain-oriented electrical steels has been well improved since N.P. Goss first proposed the technical route in 1934 [1], however, the current process is still complicated. Twin-roll casting is a novel forming process by which thin strips can be directly produced from the melt [2,3]. It has been used to manufacture lowcarbon steels and stainless steels [4–11]. The recent progress in twinroll casting has made it possible to produce grain-oriented electrical steels by a simpler way than conventional processing. For example, Liu et al. [12], Song et al. [13,14] and Wang et al. [15] have shown that the 0.23-0.27 mm thick grain-oriented electrical steel sheets can be successfully produced by a twin-roll casting route.

Secondary recrystallization is the responsible process for the formation of sharp  $\{110\} < 001 >$  preferred orientation (Goss texture) [16]. Its prerequisite is the presence of potential Goss nuclei and a fine dispersion of inhibitors in the homogeneous primary recrystallization matrix [17,18]. In conventional processing, strong Goss texture originates during hot rolling because of the intense shear deformation [19,20]. This component survives through the subsequent processing and provides the required Goss nuclei in the primary recrystallization state [21]. The desired inhibitors are mainly generated during the multi-pass hot rolling and normalizing in the acquired inhibitor method or during the complex nitriding process in the inherent inhibitor method [22-25]. By contrast, as for the twin-roll casting route, the origin of Goss texture may be greatly restricted because of the limited hot rolling reductions [26-28]. In addition, more fine and dispersed inhibitors may be generated by sufficiently controlling the precipitate evolution. Thus, the secondary recrystallization behavior may be significantly different from that in conventional processing. However, the corresponding studies have not vet been reported.

In the present paper, the microstructure and texture were characterized along the processing in a 0.23 mm-thick twin-roll cast grainoriented electrical steel. The secondary recrystallization behavior was briefly investigated. This work has led to an increased understanding of the microstructure and texture evolution during secondary recrystallization in twin-roll cast grain-oriented electrical steels.

#### 2. Experimental

The chemical composition (wt%) of the tested steel was 3.33 Si, 0.061C, 0.093 Mn, 0.026 S, 0.032 Al, 0.0075 N and balance Fe. A 3.9 mm-thick as-cast strip was produced using a laboratory vertical twin-roll caster and quenched by cold water. The experimental details of twin-roll casting are provided in Ref. [11,29]. The as-cast strip was reheated to 1130 °C, hot rolled to 2.35 mm with 39.7% reduction in two passes and cooled to room temperature. The sheets were then subjected to the normalizing in which they were first soaked at 1130 °C

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for 90 s, cooled to 930 °C in air, soaked at 930 °C for 120 s and quenched by boiling water. After this, the sheets were cold rolled to the final thickness of 0.23 mm with 90.2% reduction and subjected to primary recrystallization annealing at 830 °C for 5 min in a wet 75% H<sub>2</sub> and 25% N<sub>2</sub> atmosphere. Then, two different secondary recrystallization annealing experiments were performed, i.e. full-cycle experiment and extraction experiment. In the former one, the sheets were heated from 800 °C to 1200 °C with a heating rate of 15 °C/h and soaked at 1200 °C for 20 h. In the extraction experiment, the sheets were heated from 800 °C to 1100 °C with the same heating rate. Some samples were extracted at various temperatures from 850 °C with 25 °C interval.

The microstructures of the samples were etched with 4% nital and examined by the optical microscope. Electron backscattered diffraction (EBSD) analysis was performed to obtain the orientation image maps. The optical microscopy and EBSD were both applied on longitudinal sections as defined by the rolling direction (RD) and normal direction (ND). The deviation angles of the secondary Goss grains from the exact  $\{110\} < 001 >$  orientation were determined by EBSD mapping. The area fractions of Goss grains in the extracted samples were measured based on many optical microstructure images. The secondary recrystallization microstructures on the RD and transverse direction (TD) section were revealed by 10% hydrochloric acid. The macro-textures were examined by measuring three incomplete pole figures {110},{200} and  $\{211\}$  with CoK<sub>a1</sub> radiation in the back reflection mode by the Xray diffractometer. The orientation distribution functions (ODFs) were calculated from these pole figures by the series expansion method  $(I_{\text{max}}=22)$  developed by Bunge [30,31]. The measured layer is defined as a parameter S=2a/d, where a and d are the distances from the center and sheet thickness, respectively. In case of cubic crystal symmetry and orthorhombic sheet symmetry, it is convenient to describe the ODFs as iso-intensity diagrams in  $\varphi_2$ -sections through the Euler space. An orientation is presented in terms of the Miller indices  $\{hkl\}\langle uvw \rangle$ . Here, *{hkl}* describes the crystal plane parallel to the ND of the sheet and (uvw) is the crystal direction parallel to the RD. The most relevant fibers for the grain-oriented electrical steels are  $\lambda$ -fiber texture ( < 001 >//ND),  $\alpha$ -fiber texture (<110>//RD) and  $\gamma$ -fiber texture (<111 > //ND) which are presented in Fig. 1. The size distribution of the inhibitors was determined by a transmission electron microscope (TEM). The chemical compositions of inhibitors were verified with energy dispersive X-ray spectroscopy. The magnetic induction B8 and iron loss P<sub>1.7/50</sub> of the secondary recrystallization annealed samples sheared to 100 mm×30 mm were measured.

#### 3. Results

#### 3.1. Microstructure and texture characteristics along the processing

The microstructure of as-cast strip was a mixture of ferrite and martensite, see Fig. 2a. The ferrite matrix was characterized by outer



Fig. 1. Typical fiber textures displayed on the  $\phi_2$ =45° section.

columnar grains and inner equiaxed grains. The associated texture consisted of medium  $\lambda$ -fiber texture at the surface and random texture in the center. A striking feature was that Goss component with the intensity of 2.73 times of random distribution appeared at S=0.75 layer, which was similar with the results in [32,33]. Given that the volume fraction of martensite was below 11%, the texture of as-cast strip was mainly characterized by the orientations of initial  $\delta$ -ferrite grains just after solidification.

The hot rolled microstructure was composed of deformed ferrite grains, colonies of pearlite and fine ferrite grains and martensite, see Fig. 3a. The texture was characterized by  $\lambda$ - and  $\alpha$ -fiber texture at the surface, Goss component in the layers S=0.75-0.5 and weak  $\gamma$ -fiber texture in the center (Fig. 4a). The intensity of Goss component in the layers S=0.75 and S=0.5 was 2.78 and 2.20, respectively. After normalizing, the sheet exhibited quite similar microstructure and texture to those of hot rolled sheet. However, the normalized sheet had a more dispersive distribution of pearlite. The intensity of Goss component in the S=0.75 layer was as high as 4.93, see Fig. 4b.

After cold rolling, a severe deformation microstructure was produced, see Fig. 3c. The texture was characterized by strong  $\alpha$ - and  $\gamma$ fiber texture with the maximum at {111} < 110 > component throughout the thickness, Fig. 5a. After primary recrystallization annealing, a homogeneous microstructure consisting of fine ferrite grains was produced, see Fig. 3d. The texture was characterized by strong {111} <112 > component, medium {114} < 148 > and {001} < 120 > component, Fig. 5b. A large number of fine MnS, AlN and co-precipitates of AlN and MnS were also generated, Fig. 6. Most precipitates ranged in size from 10 to 80 nm. These precipitates may exhibit strong Zener drag effect because of their large volume fraction and small size [34].

# 3.2. Microstructure and texture evolution during secondary recrystallization annealing

Fig. 7 shows the microstructure evolution with increasing temperature in the extraction experiment. Prior to the onset of secondary recrystallization, the microstructure was composed of fine ferrite grains which exhibited a considerably low growth rate. The average size of these matrix grains only increased from 9.5  $\mu$ m at 850 °C to 11.7  $\mu$ m at 925 °C. Fig. 8 shows the image quality maps of three extracted sheets. It was found that Goss grains had no size advantage compared with matrix grains prior to the onset of secondary recrystallization, which was similar to the reports in [35,36]. This indicated that Goss grains did not exhibit higher growth rates than other matrix grains in this temperature range (850–950 °C).

In this work, 10 regions (1280 µm ×220 µm) on the RD-ND sections of each extracted sample were scanned in the EBSD measurement. The ODFs of the extracted samples were calculated based on the EBSD data. As shown in Fig. 9 and Fig. 10, in the temperature range of 850–925 °C, the texture was characterized by a strong y-fiber texture with the maximum at  $\{111\} < 112 >$  component. It was surprised that this component was gradually enhanced in the temperature range of 850-875 °C, while its intensity decreased from 900 °C to 925 °C. differing significantly from that in earlier work [37]. This may be related with the growth rate changes of matrix grains with different orientations and the shrinking of some grains for geometrical reasons. However, it was important to noted that the  $\{111\} < 112 >$  orientation at 925 °C was still stronger than that in the primary recrystallization state (Fig. 5b), in consistent with the previous reports [38-40]. The enhancement of this component may facilitate the abnormal growth of Goss grains because it exhibits a special orientation relationship with respect to Goss orientation, i.e. a rotation of 35° around <110> axis [17].

When the temperature reached up to 950 °C, secondary Goss grains appeared and grew abnormally by consuming the matrix. It was measured that the Goss grains within the matrix grew to 13.5  $\mu$ m at 950 °C and 16.7  $\mu$ m at 975 °C, while the average size of other matrix

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