



# Texture evolution of the surface layer of high silicon gradient electrical steel and influence on the magnetic properties



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## ABSTRACT

A high silicon gradient electrical steel sheet with Goss texture has been successfully prepared by a novel and simple process in this work. The FeSi alloy coating with high silicon concentration was firstly deposited on the surface of a common grain oriented electrical steel sheet substrate by cathodic arc plasma deposition technique and the coated sheet was then post-annealed at 850 °C for 6 h in a 70 % N<sub>2</sub> – 30 % H<sub>2</sub> mixed atmosphere. The results show that after annealing, an excellent Goss texture forms in the FeSi alloy coating with an average silicon concentration of about 6.5 wt. %. The high silicon FeSi alloy coating strongly influences the overall magnetic characteristics of the gradient electrical steel, and the saturation magnetizations of the grain oriented high silicon gradient electrical steel sheet along rolling direction and transverse directions of the substrate are at 251.5 and 233.0 emu/g, respectively.

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## 1. Introduction

Owing to the excellent soft magnetic properties, electrical steels are widely used as core materials in power transformers, power generators, electric motors, etc. Among the factors that influence their magnetic properties, silicon content and texture are the two critical ones. For the low silicon GO electrical steels (i.e. typically with less than 4.5 wt.% Si and {110}<001> Goss texture), the formation of Goss texture is very complicated, the main manufacture processes include hot rolling, cold rolling, annealing, decarburizing, box annealing, etc., the final textures and microstructures are determined by each processing step [1]. The reduction of cold rolling varies from 55 % to 87 % depending on different cold rolling processes (i.e. single or two stage cold rolling processes), during which the deformation textures mainly composed of {111}<112> can form [2]. The inhibitors or impurities which can nail the grain boundaries and retard the movement of boundaries of the grains with other orientations except {110}<001> during the secondary recrystallization annealing are very important for the abnormal

growth of Goss grains and the formation of strong Goss texture [3–5]. In brief, three necessary conditions are met simultaneously for the formation of Goss texture in the common low silicon GO electrical steels, i.e. Goss nuclei with a size advantage, inhibitors and cold rolling deformation textures mainly composed of {111}<112> texture [2–7]. In respect of theoretical studies, two main models, i.e. coincidence site lattice (CSL) [8] and high-energy boundary (HE) [9], have been used to explain the mechanisms about the abnormal Goss grain growth till now.

High silicon electrical steels, particularly the ones with 6.5 wt.% Si and Goss texture, possess much better combination of magnetic properties than low silicon GO ones [10–13]. Consequently, more and more attentions are gradually focused on how to obtain Goss texture in Fe – 6.5 wt.% Si electrical steel but the improvement of the textures stays in contrast with the lack of ductility [14]. Even through the Fe – 6.5 wt.% Si electrical steel with Goss texture could be prepared by secondary recrystallization technique but no details were given [13]. And then, extensive studies have been made on the texture evolution in 6.5 wt.% Si electrical steel. It is found that alloying elements, producing technologies, process conditions, etc., strongly influence the texture evolution of the Fe – 6.5 wt.% Si steel. Many different types of textures can be obtained in Fe – 6.5 wt.% Si based alloy depending on different producing methods, process

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conditions, alloying elements and heat treatments [15–25]. Ros-Yáñez et al. [19–21] found that the Fe – 6.5 wt.% Si steel prepared with hot dipping process in a hypereutectic Al – 27 wt.% Si molten bath exhibited an ideal ND fiber toward  $\{554\}\langle 225 \rangle$  and  $\{110\}\langle 110 \rangle$  components after annealing; an intense  $\gamma$  fiber, a weak cubic fiber or a strong  $\gamma$  fiber was developed, respectively, depending on intermediate cold rolling (ICR) reductions. As for the Fe – 6.5 wt.% Si – 1 wt.% Al alloy prepared with spray forming and warm rolling technique, the initial texture  $(011)[0\bar{1}1]$  is changed to approximately  $(013)[1\bar{1}0]$  after the last reduction step, and the entire texture finally changes to a partial ND fiber running from  $\{001\}$  to  $\{110\}$  with negligible Goss intensities after warm-rolling [22]. Similar to the low silicon GO electrical steels, the Goss texture dominates the superficial layer of the hot rolled Fe – 6.5 wt.% Si sheet, and a strong  $\gamma$  fiber appears after cold rolling, the texture transforms to  $\eta$  fiber with a peak at  $\{210\}\langle 001 \rangle$  after primary recrystallization [24]. Wang et al. [25] found that the Goss nuclei formed in the Fe – 6.5 wt.% Si – 0.3 wt.% Al thin sheet produced by strip casting and warm rolling at the early stage of recrystallization but failed to grow due to the marked disadvantage in size and number; instead,  $\lambda$  fiber dominated the recrystallization texture while  $\gamma$  fiber almost disappeared after annealing.

These results demonstrate that cold rolling with heavy reduction is hard to be implemented on the Fe – 6.5 wt.% Si binary alloy, even though its ductility could be markedly increased through adding Al, B, etc., and more than 80% cold rolling reduction can be obtained, the above mentioned conditions for the formation of Goss texture in the low silicon GO electrical steel is hardly met in the Fe – 6.5 wt.% Si based alloy, that is, stable Goss nuclei, inhibitors and cold rolling deformation textures mainly composed of  $\{111\}\langle 112 \rangle$  texture, which can turn to Goss texture by rotating around  $\langle 110 \rangle$  parallel to the transverse direction. Consequently, the texture evolution is very complicated and the Goss texture is very difficult to be obtained.

In this work, a novel and simple method is attempted to obtain high silicon gradient electrical steel sheet with Goss texture. In this method, a high silicon FeSi alloy coating is firstly deposited on both sides of a commercial low silicon GO electrical steel sheet by cathodic arc plasma deposition technique, and the coated steel sheet is then annealed. The texture evolution of the surface FeSi alloy coating with high silicon content is studied and its mechanism is also discussed.

## 2. Experimental procedure

The high silicon gradient electrical steel sheet studied in this work was prepared by cathodic arc plasma deposition technique. The target was a Fe – 7.5 wt.% Si alloy prepared by traditional vacuum melting method with iron and silicon of a purity of 99.90% and 99.95%, respectively; the GO electrical steel substrate was a kind of 0.27 mm thick commercial grade products manufactured by Wuhan Iron and Steel Group Company Limited (WISCO), i.e. 27Q130. Firstly, the insulation layer on the surface of the GO electrical steel sheets was peeled thoroughly, and then their surface was polished carefully; after that, the sheets were cut into pieces with a size of 30 mm  $\times$  30 mm, followed by ultrasonically cleaned in ethanol and acetone for 10 – 15 min, respectively. The substrates were then loaded into the sputtering chamber after being blow-dried with pure  $N_2$  gas. Before deposition, the target and substrates were cleaned with glow discharge of Ar with a purity of 99.999% after the base pressure of the chamber was pumped down to lower than  $5.0 \times 10^{-2}$  Pa. The deposition process was carried out on condition that the flow rate of high purity Ar of 99.999%, the sputtering pressure, sputtering current and deposition time were controlled at 18.0 sccm, 2.8 Pa, 80–85 A and 2–4 h, respectively.

After deposition, a portion of the specimen together with a portion of FeSi alloy coating peeled from the substrate were heated together from room temperature to 850 °C and 1200 °C, respectively at a rate of about 200 °C/h and soaked for 6 h; and then, they were cooled down to room temperature in the furnace. During the whole heat-treatment process, all the specimens were protected by a 70 %  $N_2$  – 30 %  $H_2$  mixed atmosphere.

Since there are two types of X-ray source for X-ray diffraction apparatus, the phases of the specimens were identified by X'pert MPD Pro XRD with line shaped X-ray source while the  $\{200\}$ ,  $\{110\}$  and  $\{111\}$  pole figures of the as-deposited and post-annealed specimens were measured by PANalytical Empyrean XRD with spot shaped X-ray source, and  $CuK\alpha$  radiation ( $\lambda = 0.15406$  nm, 40 kV, 40 mA) was used for the measurement. After that, the orientation distribution figures (ODFs) and grain boundary character distribution calculation were deduced from these pole figures. The surface morphologies and cross-section of the specimens were observed by FEI Nano 400 field emission scanning electron microscope, and selected-microarea elemental analysis was carried out by energy-dispersive x-ray spectroscopy (EDS, IE350 Penta FET X-3) attached to the SEM. The local textures of the specimen required electron backscattered diffraction (EBSD) were achieved using FEI Nano 400 SEM equipped with HKL Channel5 EBSD.

The magnetic properties were characterized using a vibrating sample magnetometer (VSM, Model JDAW-2000) at 25 °C under an external magnetic field with a maximum value of 5000 Oe applied parallel to the rolling direction (RD) and transverse direction (TD) of the substrate, respectively, the demagnetizing factor was 0.33 and the specimen size was 4 mm  $\times$  4 mm.

## 3. Results and discussion

The SEM images of the surface morphologies and cross-section of the as-deposited and post-annealed (at 850 °C) specimens and their silicon concentration depth profiles are shown in Fig. 1. The as-deposited FeSi alloy coating with a thickness of about 10–15  $\mu$ m clings closely to the substrate surface while the coating-substrate interface is clear (Fig. 1a), the average Si concentrations of the FeSi alloy coating and substrate are stable at about 7.89 wt.% and 3.5 wt.%, respectively, indicating that almost no Si diffusion occurred between the coating and substrate. After annealing at 850 °C, the Si atoms obviously diffused from the high silicon FeSi alloy coating into the common low silicon GO electrical steel substrate caused by the large difference in Si concentration between them; consequently, the Si concentration in the FeSi alloy coating decreases to about 6.5 wt. % and gradually declines with depth. On the contrary, the Si concentration in the substrate slightly increases to about 3.9 wt. % at a depth of about 5  $\mu$ m and is almost stable at this degree in a range of 5–25  $\mu$ m, which is higher than the original concentration. Furthermore, post-annealing and diffusion also lead to an analogous metallurgical bonding between the FeSi alloy coating and substrate. The SEM images of the surface morphologies show that the as-deposited FeSi alloy coating is composed of compact uneven-sized spherical particles with fuzzy boundaries and there exist some teeny-weeny superficial holes. In contrast, the post-annealed FeSi alloy coating is composed of analogous hexagonal homogeneous grains and most of their boundaries are very sharp. These results confirm that no abnormal grain growth occurs in the FeSi alloy coating post-annealed at 850 °C.

Since preferred orientation may exist in the specimens, especially in the GO electrical steel substrate, the XRD measurements for the phase analysis using line shaped X-ray source were made in two directions, i.e. the line shaped X-ray source was parallel to the RD and TD of the GO substrate and the corresponding XRD spectra were then named as RD and TD, respectively, as shown in Fig. 2. Due

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