



## Research articles

## Effects of rolling conditions on recrystallization microstructure and texture in magnetostrictive Fe-Ga-Al rolled sheets

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## ARTICLE INFO

## Article history:

Received 28 September 2017

Received in revised form 26 February 2018

Accepted 26 February 2018

Available online 27 February 2018

## Keywords:

Magnetostriction

Fe-Ga-Al alloy

Rolling

Secondary recrystallization

Goss texture

## ABSTRACT

The effects of different rolling conditions on the microstructure and texture of primary and secondary recrystallization in magnetostrictive  $\text{Fe}_{82}\text{Ga}_9\text{Al}_9+0.1\text{at}\%\text{NbC}$  alloy sheets were investigated. After the primary recrystallization annealing at  $850^\circ\text{C}$  for 5 min, the as-rolled sheets prepared by warm-cold rolling with an intermediate annealing, can be fully recrystallized, and obtain the homogeneous matrix in which the fine dispersed NbC precipitate particles are distributed. The primary recrystallization textures of sheets with different rolling conditions consist mostly of strong  $\{1\ 0\ 0\}$  textures,  $\gamma$ -fiber textures,  $\{4\ 1\ 1\}$   $\{1\ 4\ 8\}$  texture and weak Goss texture. In the primary recrystallized sheets prepared by warm-cold rolling with an intermediate annealing, the high energy grain boundaries and  $\Sigma 9$  boundaries have the highest proportion. After high temperature annealing, the secondary recrystallizations of Goss grains in these sheets are more complete, and the size of abnormal grown Goss grain is up to several centimeters, which results in the strongest Goss texture. Correspondingly, the largest magnetostriction of 183 ppm is observed. The sample prepared by warm-cold rolling with an intermediate annealing, has homogeneous primary matrix, special texture components and grain boundary distribution, all of which provide a better surrounding for the abnormal growth of Goss grains. This work indicates that the control of rolling conditions of Fe-Ga-Al alloy sheets is necessary to achieve the strong Goss texture and obtain a possible high magnetostriction if other appropriate conditions (stress, domain structure) are achieved.

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## 1. Introduction

Magnetostrictive material is one of the most important magnetic functional materials, and can be applied to actuator, sensor, and energy harvesting [1]. Because of the eddy current losses, it is generally hoped that magnetostrictive alloys can be used in the form of thin sheets rather than bulks when used for high-frequency electromagnetic fields. Fe-Ga and Fe-Al alloys, as two kinds of main magnetostrictive alloys, have their own special features and are widely studied [2–8]. On one hand, the maximum saturation magnetostriction of single crystal Fe-Ga alloys is up to 400 ppm, but polycrystalline Fe-Ga alloys are easily to crack during rolling processes due to weak grain boundaries [2,3]. On the other hand, the ductility and machinability of Fe-Al alloys are shown better than Fe-Ga alloys, however, the maximum saturation magnetostriction of single crystal Fe-Al alloys is only 140 ppm [4,5]. Recently, our previous work has indicated that the  $[1\ 0\ 0]$  oriented

polycrystalline alloy of  $\text{Fe}_{82}\text{Ga}_9\text{Al}_9$  exhibited a fracture elongation of 16.5% at room temperature and a maximum saturation magnetostriction of  $\sim 200$  ppm without pre-compressive stress, indicating the good ductility and magnetostrictive property [9]. This combination of magnetic and mechanical properties makes Fe-Ga-Al alloy an appropriate material for magnetostrictive rolled sheets and wires produced by the conventional thermo-mechanical processing approach.

As the largest saturation magnetostriction coefficient in single crystal Fe-Ga and Fe-Al alloys are both obtained along  $\langle 1\ 0\ 0 \rangle$  direction, besides phase structure and magnetic domain configuration, the development of strong  $\langle 1\ 0\ 0 \rangle$  orientation is also critical to achieve maximum magnetostriction in Fe-Ga-Al alloy sheets. In grain-oriented silicon steel which has a similar structure with Fe-Ga(Al) alloys, Goss  $\{1\ 1\ 0\}\langle 0\ 0\ 1 \rangle$  and cubic  $\{1\ 0\ 0\}\langle 0\ 0\ 1 \rangle$  textures are desirable to get high magnetic flux density and low core loss, because  $\langle 0\ 0\ 1 \rangle$  direction is the easy magnetization axis. Although the cubic-oriented silicon steel has captured the interests of many researchers because of its symmetrical simplicity and the beneficial influence on magnetic properties, cubic texture hardly evolves

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in bcc (body-centered cubic) Fe-Si alloy by ordinary rolling and annealing processes [10]. By contrast, the Goss texture develops relatively with ease upon secondary recrystallization annealing after rolling, and the theoretical research and industrial production of Goss-oriented silicon steel has been very mature. Referring to the grain-oriented silicon steel, developing Goss texture through secondary recrystallization is considered as the most effective way to achieve the sharp  $\langle 001 \rangle$  orientation in the rolled Fe-Ga and Fe-Al alloy sheets, and many related works have been reported [11–15]. During the secondary recrystallization, the grain growth of primary recrystallization is restrained by strong pinning effect from the fine and dispersed inhibitors, and Goss textured grains can break the pinning effect and achieve the abnormal growth. Thus, the conventional thermo-mechanical processing and texture annealing are regarded as a feasible way to prepare  $[100]$  textured materials at low cost compared to the single crystal and directional solidified materials. As well known, the production processes of Goss grain-oriented silicon steel is complicated, which generally includes steps of melting, continuous casting, hot rolling, normalizing annealing, cold rolling, decarburizing annealing, final high temperature annealing, etc. [16]. Among which, it is very important that the primary recrystallization takes place to form some Goss grains with accurate orientation and a suitable matrix structure during the decarburizing annealing, which benefits the subsequent abnormal growth of Goss grains to achieve the strong Goss texture. For Fe-Ga alloy sheets, many previous studies mainly focused on the effects of high temperature annealing conditions on the secondary crystallization, which generally includes anneal temperature, time, heating rate, atmosphere, etc [11,17,18]. Recently, the influences of intermediate annealing and strong NbC particle pinning on promoting abnormal growth of Goss grains in the rolled  $\text{Fe}_{82}\text{Ga}_{4.5}\text{Al}_{13.5}$  alloys were reported [19,20].

Rolling process is a critical step in the production of grain-oriented silicon steel, because of the effects on the primary and secondary recrystallization. In the work of Na et al. [21,22], individual solid NbC particles used as inhibitors were added to the melt and stirred sufficiently to be uniformly dispersed in the Fe-Ga matrix while melting. However, in systems with precipitated particles, it is important to control rolling process, mainly including the rolling temperature, reduction rate, intermediate annealing (as named normalizing annealing in grain-oriented silicon steel), etc, because the rolling conditions not only impact the primary microstructure and texture, but also deeply influence the precipitation behavior of inhibitor particles, which are all very important for secondary recrystallization. The reports about effects of rolling process on recrystallization microstructure, texture and magnetostriction of Fe-Ga-Al alloy sheet are rarely seen. In this work, a Goss grain-oriented Fe-Ga-Al alloy sheet was prepared, and different rolling conditions were chosen to investigate their effects on the microstructure and texture of primary and secondary recrystallization, which could provide theoretical basis for the formulation of rolling process in magnetostrictive Fe-Ga-Al alloy sheets.

## 2. Experimental

The alloys with nominal composition of  $\text{Fe}_{82}\text{Ga}_9\text{Al}_9+0.1\text{at}\%\text{NbC}$ , were prepared from Fe (99.9%, weight percent), Ga (99.99%, weight percent), Al (99.9%, weight percent), and master alloys of Nb-Fe and Fe-C. A columnar-grained ingot with  $[001]$  preferred orientation was produced by the directional solidification technique at a growth rate of  $720\text{ mm}\cdot\text{h}^{-1}$ . A detail description of the directional solidification process could be found in our previous work [23]. Some slabs were cut from the directional solidified ingot by electrical discharge machining, and then were rolled to the final thickness of  $\sim 0.3\text{ mm}$  by different rolling processes. The long axes of columnar grains were arranged along rolling direction (RD) during rolling process. The detailed rolling conditions could be found in Table 1. The samples which underwent different rolling processes were indicated as sample A, B and C. The as-rolled sheets, with size of  $0.3\text{ mm} \times 12\text{ mm} \times 16\text{ mm}$  cut by electrical discharge machining, were enclosed in quartz ampoules using 0.3 atm. Ar as protecting gas. The sheets enclosed in the ampoules were annealed at  $850^\circ\text{C}$  for 5 min for primary recrystallization. After the primary annealing, samples were rapidly heated from  $850$  to  $900^\circ\text{C}$  with a rate of  $10^\circ\text{C}/\text{min}$  in the furnace, and then were slowly heated from  $900$  to  $1080^\circ\text{C}$  at a controlled rate of  $1.0^\circ\text{C}/\text{min}$  without dwell at  $1080^\circ\text{C}$ . The final annealing process was undertaken at  $1200^\circ\text{C}$  for 6 h under flow  $\text{Ar}/\text{H}_2$  ( $\text{H}_2\text{vol}\% = 25\%$ ) mixed atmosphere, followed by furnace cooling to around  $850^\circ\text{C}$  and air cooling to room temperature.

Microstructure of the directional solidified ingot was observed by optical microscope (OM) and scanning electron microscope (SEM), and energy dispersive spectroscopy (EDS) was employed to identify the composition of precipitates. The microstructures, grain configurations and orientation distributions of the recrystallized sheets were determined by electron backscatter diffraction (EBSD) system. The distributions of precipitate particles were observed by SEM. The SEM, EDS and EBSD were carried out on a SUPRA™ 55 field emission scanning electron microscope. The magnetostriction was measured by strain gauge, and the gauges were positioned along the rolling direction. For the magnetostriction measurement ( $\lambda_{\parallel}$  and  $\lambda_{\perp}$ ), a magnetic field parallel and perpendicular to the rolling direction was applied respectively. The apparent saturation magnetostriction was calculated by  $(3/2)\lambda'_s = \lambda_{\parallel} - \lambda_{\perp}$ .

## 3. Results and discussion

The longitudinal optical microstructures of directional solidified  $\text{Fe}_{82}\text{Ga}_9\text{Al}_9+0.1\text{at}\%\text{NbC}$  alloy is shown in Fig. 1. From OM images (Fig. 1(a)), it can be found that the grain growth direction is approximately parallel to the solidification direction, and the size of a single columnar crystal is very big. Additionally, a large amount of precipitates, most with bar-shaped and size up to  $20\text{--}30\text{ }\mu\text{m}$ , are distributed both in the grains and at grain boundaries, as shown in Fig. 1(b). EDS were employed to get further identification of precipitates. The result shown in Fig. 1(c) demonstrates that the precipitates are identified as niobium carbide (NbC).

**Table 1**  
Rolling conditions of  $\text{Fe}_{82}\text{Ga}_9\text{Al}_9+0.1\text{at}\%\text{NbC}$  alloy sheets.

Samples	Rolling conditions							
	Hot rolling		Warm rolling		Intermediate annealing		Cold rolling	
	Temperature/ $^\circ\text{C}$	Thickness change/mm	Temperature/ $^\circ\text{C}$	Thickness change/mm	Temperature/ $^\circ\text{C}$	Time/min	Temperature/ $^\circ\text{C}$	Thickness change/mm
A	1150	18.0–2.0	600	2.0–1.0	850	5	Room temperature	1.0–0.3
B	–	–	600	8.0–1.0	850	5	Room temperature	1.0–0.3
C	–	–	600	8.0–1.0	–	–	Room temperature	1.0–0.3

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