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Characterizing microstructure and texture after recrystallization annealing of Hi-B steel with simultaneous decarburization and nitriding

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ABSTRACT

How to manufacture the high magnetic induction grain-oriented silicon steel (Hi-B steel) by the process featured with the primary recrystallization annealing was demonstrated, during which nitriding and decarburizing were simultaneously realized in laboratory. By the techniques of optical microscope, scanning electronic microscope and electron backscattered diffraction, both the microstructure and the texture in the samples were characterized. The samples had been subjected to nitriding to different nitrogen contents at two specified temperatures using the two defined microstructural parameters: the grain size inhomogeneity factor σ^* and the texture factor AR. The former is the ratio of the mean value to standard deviation of grain sizes; the latter is the ratio of the total volume fraction of the harmful textures to that of beneficial textures including {110}<001>. When the N content increased from 0.0055% to 0.0330% after the annealing at both 835 and 875 ℃, the resultant recrystallized grain size decreased but σ^* changed little; whilst the rise of annealing temperature from 835 to 875 ℃ resulted in the increase in both grain size and σ^*. Moreover, either the injected N content or temperature had insignificant influence on the components of primary recrystallization texture developed during annealing. However, the increase of temperature led to the decreases in both intensity and volume fraction of {001}<120> and {110}<001> textures but increases in the {114}<481> and γ fiber textures and the resultant decrease of AR.

1. Introduction

Compared with traditional high-temperature (1350-1 400 ℃) heating method, the low-temperature and medium-temperature reheating method are development direction of producing Hi-B steel[1-4]. In this technology, nitriding is a key process to form nitride as inhibitors for the development of sharp Goss texture during high temperature annealing[5-7].

There are several nitriding technologies developed for Hi-B steel in the commercial or the trial industrial production lines. For examples, Bao Steel, WISCO and Shougang Group in China and Nippon Steel in Japan all employed the process of nitriding after deccarburization during recrystallization annealing in the atmosphere containing NH_3[4,7]. However, POSCO adopted a simultaneous nitriding and decarburizing process during annealing. In comparison with the decarburization followed by nitriding, the simultaneous nitriding and decarburizing has some advantages such as the more compact process and lower production cost of nitriding etc.[8,9]. Although there are some reports about inhibitors formed during this simultaneous process[8-10], few researches are about the dependence of primary recrystallized microstructure and texture on the annealing process.

In this paper, the Hi-B steel was manufactured from the thin slab casting, the low-temperature reheating, rolling process and the simultaneous nitriding and decarburizing annealing to the final high-temperature annealing. Both microstructure and texture after primary recrystallization were characterized and analyzed using the defined microstructural parameters: the grain size inhomogeneity factor and the texture factor. It will provide references on researching Hi-B steel by obtaining inhibitor method at low-temperature nitriding.

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2. Experimental Materials and Methods

2.1. *Preparation of experimental materials*

The chemical composition (wt. %) of studied steel is C 0.04－0.06, Si 2.90－3.30, Mn 0.2, Cu 0.02, S 0.0047, Al_s 0.029, N 0.0055, and P 0.02. It was melted by vacuum induction furnace and then poured into a water-cooled copper-made mould with the dimensions of 50 mm×100 mm×400 mm. The cast slab was then transferred from the mould at the temperature higher than 950 ℃ to a box furnace, in which it was heated at 1180 ℃ and held for 30 min. Later, it was hot rolled to 2.6 mm-thick strip and then cold rolled to 0.27 mm by the single-stage cold-rolling after the two-step normalization. Subsequently, the specimens cut from cold rolled strips were subjected to the simultaneous nitriding and decarburization annealing at both 835 and 875 ℃ for 3 min in the mixture gas of $NH_3+H_2+N_2$, in which NH_3 is 1.2－7.0 vol. % and V_{H_2}/V_{N_2} is 1/3 for achieving different N contents injected into specimens. Finally, these specimens underwent the high temperature annealing during which the sharp Goss texture was developed. The magnetic properties of these finally annealed specimens with the dimension of 30 mm × 100 mm were measured by MATS-2010SA exchange magnetic measuring instrument. The average values of B_8 and $P_{17/50}$ magnetic properties with the primary recrystallization annealing at 835 and 875 ℃ were measured as 1.921 T/0.968 W/kg and 1.935 T/0.945 W/kg, respectively, and the best values are 1.964 T/0.854 W/kg and 1.945 T/0.878 W/kg, respectively.

2.2. *Observation and measurement of microstructure*

The microstructure of nitriding sheet through thickness was observed and captured by ZEISS-Axio Scope A1 optical microscope at 100 magnifications. According to line resection in average grain size determination (GB/T 6394-2002) of metal, the average grain size $\overline{X}$ of microstructure of nitriding sheets was measured. 3－4 fields were randomly selected, and the number of cutoff points was about 500 in order to obtain the reasonable and accurate average values.

In addition, the inhomogeneity of grain size σ^* is defined as the ratio of the standard deviation to the mean value of grain size, as given as follows.

$$\sigma^* = \sigma/\mu \tag{1}$$

where

$$\sigma = \sqrt{\frac{1}{N}\sum_{i=1}^{N}(X_i-\mu)^2} \tag{2}$$

σ is the standard deviation of grain size; N is the quantity of grains; X_i is the size of single grain, μm; and μ is the average size of all the grains in total microstructure, μm. σ is a reflection of deviation degree of the single grain size in total primary grains' microstructure away from μ. The higher value of σ^* means greater inhomogeneity of grain size formed after the recrystallization annealing.

2.3. *Examination of texture*

The textures of samples after recrystallization annealing through thickness were examined by field emission scanning electron microscope, ZEISS SUPRA 55VP, with electron backscattered diffraction (EBSD). At least 2 regions in each sample were scanned with the step size of 3 μm.

The software of texture, OIM Analysis 6.1, was used in analyzing the orientation distribution function (ODF), the distribution and quantitative statistics of grain with specified orientation with the maximum deviation angle of 10°. At the same time, the grain sizes were analyzed statistically to obtain the values of μ and σ.

The texture factor, AR (area ratio), was defined to quantitatively characterize the textures after the primary recrystallization. AR is the ratio of the total volume fraction of the harmful textures to that of beneficial textures including {110}<001>. The lower value of AR means lower ratio of the disadvantaged texture in primary recrystallization texture, and higher possibility of abnormal growth of Goss oriented grain.

Some researches show that the textures like {111}<112> and {411}<148> favor the growth of Goss grains[11-14]; whilst the α fiber textures, including {110}<112>, {111}<110> and {112}<110> etc. are deteriorating[15-18]. Therefore, the texture factor AR is defined as follows:

$$\mathrm{AR} = (\{hkl\}\langle110\rangle + \{110\}\langle112\rangle) / (\{111\}\langle112\rangle + \{554\}\langle225\rangle + \{114\}\langle481\rangle + \{110\}\langle001\rangle) \tag{3}$$

where, $\{hkl\}\langle110\rangle$, the α fiber texture, is the total fraction of {001}<110>, {114}<110>, {112}<110>, {111}<110> and {110}<110>; $\{111\}\langle uvw\rangle$, the γ fiber texture, is the total fraction of {111}<110>, {111}<112> and {554}<225>, and the last one is included since it is close to {111}<112>.

3. Results

3.1. *Microstructure characteristic of nitriding sheet*

The simultaneous nitriding and decarburizing annealing temperatures of the cold rolled sheets are 835 and 875 ℃. When there is low scale of NH_3, the steel can reach enough nitriding content. That is to say, the nitrous infiltration becomes easy. At the same time, residual carbon content in steel remains low (0.0008－0.0027 wt. %), and thus the decarburizing is easy. The