

## Simultaneous decarburizing and nitriding: effects on structure and magnetic properties of 3%Si grain-oriented electrical steel

<http://dx.doi.org/10.1590/0370-44672017710076>

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### Abstract

Lab experiments were carried out to check the effect of some process parameters of nitriding, performed simultaneously with decarburization, on magnetic properties of 3%Si Grain-oriented silicon steel produced by a low slab reheating technology. The samples of (GO) 3% Si steel collected as 0.27mm cold-rolled were submitted to a continuous annealing process at two temperatures, 860 and 900°C; three annealing times, 63.6, 95.4, and 119.25s; and three ammonia flows 0.1, 0.2, and 0.4Nm<sup>3</sup>/h. The nitrogen content obtained after the treatments was much higher than those obtained by the currently used process, and it increased as the ammonia flow rates and soaking times were increased, hindering the primary grain growth that adversely affected the magnetic properties. The increase of annealing temperature improved the decarburization and the magnetic properties. The best magnetic properties were found in the sample with the highest primary grain size and the lowest amount of absorbed nitrogen. The success of this practice, in terms of magnetic properties, was pointed out to be based on a drastic reduction on strip nitrogen increase.

**Keywords:** electrical steels; grain-oriented; simultaneous decarburization and nitriding.

### 1. Introduction

Grain oriented electrical steel can be manufactured using low slab reheating temperature technology with (Al,Si)N as the main grain growth inhibitor to promote secondary recrystallization during box annealing treatment. In this acquired inhibitor technology, the precipitates can be formed by the strip nitriding with NH<sub>3</sub> after decarburization annealing. Decarburization and nitriding are performed into two different chambers separated by sealing devices. In this case, nitriding

occurs after decarburization and primary recrystallization, allowing a better control of the primary grain size, an important microstructural parameter that affects the secondary recrystallization process and the final magnetic properties (Kumano *et al.*, 2005; Xia *et al.*, 2008; Liao and Hou, 2010).

In this study, decarburization and nitriding were carried out simultaneously in the same section, to check the feasibility of this process in order to simplify

the equipment and operation practices. The parameters of temperature, ammonia flow and time were varied in order to show their effects on microstructure and nitrogen content in the strip, their consequences on the final structure and magnetic properties of the steel, and verification if good magnetic properties could be achieved by acting on these parameters. A summary of the results of this research and the suggestions to achieve better magnetic results are presented.

## 2. Material and method

Industrial cold-rolled 0.27mm grain-oriented 3.2% Si samples was obtained by a low slab reheating temperature-1150°C and Steckel reversible finishing mill. They were subject to hot band annealing at 1000°C/90s; cooled down in two stages at 7°C/s to 900°C; quenched with 40°C/s to room temperature; and warm rolled at 200°C in the 2<sup>nd</sup> and 3<sup>rd</sup> passes, whose chemi-

cal composition is shown in Table 1. They were also processed in the Lab to simulate simultaneous decarburization and nitriding processes by performing a continuous annealing with an atmosphere of 0.5 Nm<sup>3</sup>/h N<sub>2</sub> – 0.5 Nm<sup>3</sup>/h H<sub>2</sub> with the dew point 60°C, at two temperatures 860 and 900 °C, three annealing soaking times 63.6, 95.4, and 119.3s and three ammonia flows

0.1, 0.2, and 0.4Nm<sup>3</sup>/h. Afterwards, the samples were coated with MgO and treated in a box annealing furnace, where they were heated at 100°C/h from room temperature up to 400°C, and at 10°C/h from 400°C up to 1200°C in 75%H<sub>2</sub> + 25%N<sub>2</sub> dry atmosphere. They were kept at 1200°C for 15h in an atmosphere of 100% H<sub>2</sub> and cooled down to the room temperature.

C	Mn	S	Si	AlS	Altotal	Cu	Sn	N
0.049	0.06	0.008	3.18	0.025	0.028	0.4	0.08	0.0064

Table 1  
Chemical composition of steel (weight%).

Characterization of the microstructure after decarburization and nitriding was made using optical (OM) and scanning electron microscopy (SEM). Chemical analyses of carbon were made using the Leco-CS600 apparatus, and oxygen and nitrogen using Leco TC436 device. The primary grain

size was measured by the free intercept method using Electron Back-Scattering Diffraction (EBSD) with 1µm step and 15° tolerance angle, and the secondary grain size was measured manually by the equivalent diameter method. The average Goss orientation deviation texture was obtained by EBSD using

a stacking of samples, in order to get a reasonable number of grains being analyzed. Magnetic tests in the longitudinal direction were performed in a MPG10D device (Brockhaus manufacturer) with strips of 100 mm x 30mm measuring core loss at 1.7T/60 Hz and magnetic induction at 800 mA/60 Hz.

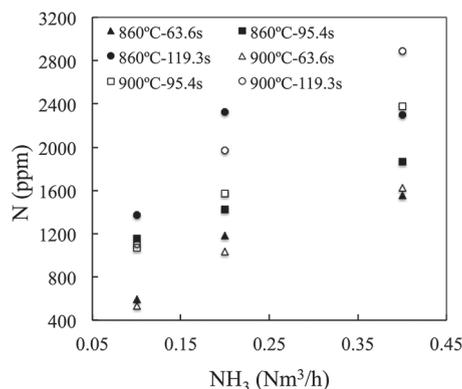
## 3. Results and discussions

### Chemical Composition

Nitrogen content in the strip increased when ammonia flow and annealing time increased. Temperature effect on nitrogen content is more expressive for the longer time, as can be seen in the Fig.1. Furthermore, Nitrogen content obtained in all the samples was extremely high in comparison with the standard process whose value varies from 150 to 300ppm (Lee *et al.*, 2002; Joo *et al.*, 2008). The nitrogen content in the sample nitrated for 63.6s with ammonia flow of 0.1Nm<sup>3</sup> was approximately 533ppm at anneal-

ing temperature of 860°C. However, nitrogen content in the sample that was nitrated for 119.3s at 900°C with ammonia flow of 0.4Nm<sup>3</sup> reached 2885ppm. The higher soaking time and the higher ammonia flow inside the treatment chamber contributed for such achieved high level of nitrogen, in comparison to the standard processes. The presence of higher humidity that forms a more permeable oxide layer also contributed for the nitrogen content increasing.

The nitriding atmosphere used in



this study was a mixture of hydrogen, nitrogen and ammonia gas and humidity. At temperature in the range of 500-550°C, the ammonia gas underwent the cracking process and dissociated into nitrogen and hydrogen atoms on the surface of the strips. Thus, the nitrogen freshly generated in this process was subsequently absorbed into the strips by the diffusing through the surface. It can be observed that the nitrogen amounts in the strips increased with increasing nitriding time and ammonia flow (Liao and Hou, 2010).

Figure 1  
Influence of the ammonia flow on strip nitrogen content at 860 and 900°C in different treatment times.

The ammonia flow did not influence the carbon content obtained. An acceptable carbon content level in the strip is C ≤

30ppm (Bernier *et al.*, 2013), a value that was only reached for 119.3s and 95.4s times (Fig. 2). The effectiveness of the decarbu-

rization was achieved for all the treatment times at 900°C, but not for the shortest time at 860°C, as can be seen in the Fig. 3.

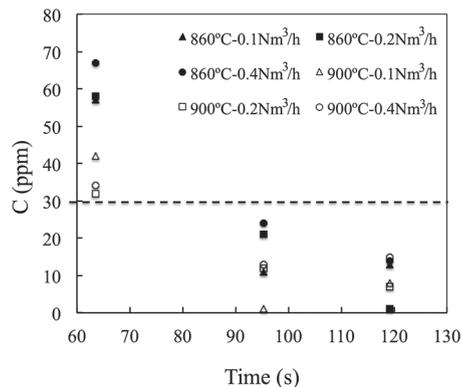


Figure 2  
Influence of the treatment time on carbon content at 860 and 900°C in different ammonia flows.

**Structure**

The increase on strip nitrogen content decreased the primary grain size, while the treatment at 900°C produced the larger primary grain size, mainly for a lower nitrogen content (Kumano et al., 2005; Xiang et al. 2017), as shown in the Fig. 3. The mean primary grain

size varied from 8 to 10µm. This small grain size was a consequence of the nitration occurring simultaneously with the recrystallization and grain growth, that was more intense, forming a big amount of precipitates and causing the nitrogen content obtained in all the samples to be

extremely high in comparison with the standard process (from 150 to 300ppm) (Lee et al., 2002; Joo et al., 2008). These phenomena happening together inhibit the grain growth in an earlier stage, when the proper primary grain size is not yet achieved.

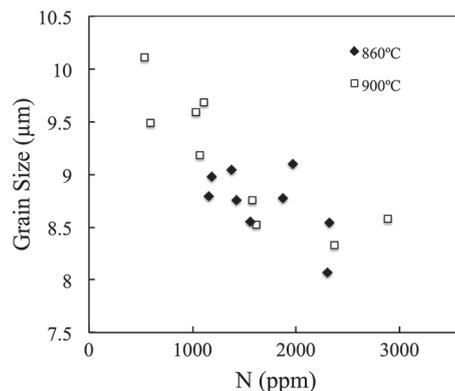


Figure 3  
Influence of the strip nitrogen on the primary grain size at 860 and 900°C.

Fig. 4 shows the microstructure of samples recrystallized under different annealing conditions and there can be ob-

served a slight difference in size between the grains close to the surface and close to the centre of the samples. The grains close to

the surface are smaller than those close to the centre due to more intense precipitation that occurred on the steel surface (Fig. 5).

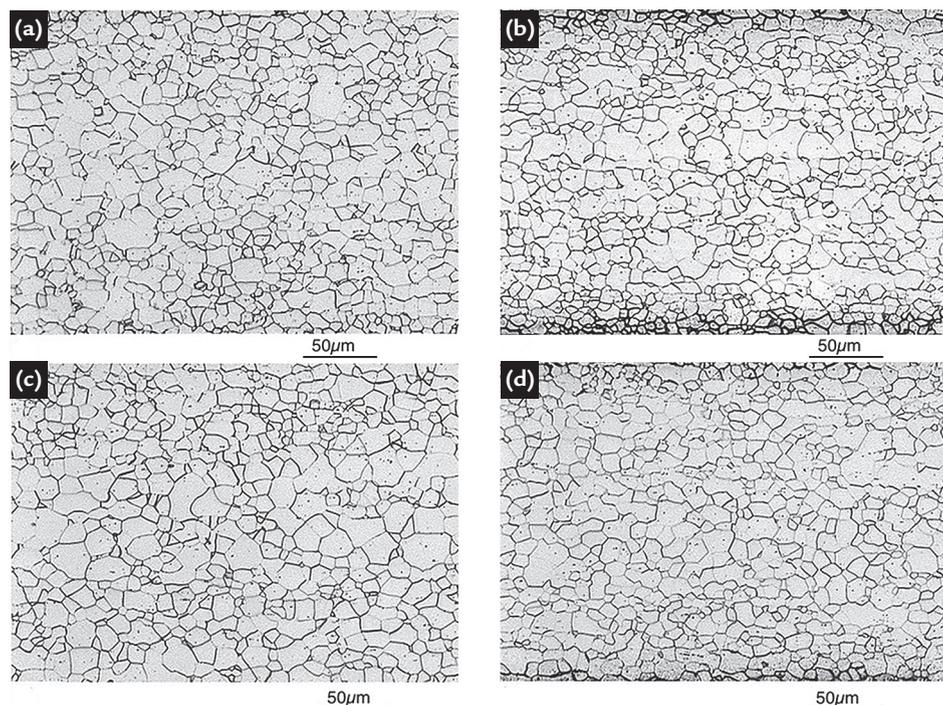


Figure 4  
Micrographs of samples after decarburization and nitriding (temperature, time, ammonia flow): a) 860°C, 63.6s, 0.1Nm³/h; b) 860°C, 95.4s, 0.4Nm³/h; and c) 900°C, 63.6s, 0.1Nm³/h; d) 900°C, 95.4s, 0.4Nm³/h. OM.

Fig. 5 shows a region of approximately 25µm from the surface with a high concentra-

tion of precipitates, and there can be observed an increase of precipitate volume fraction

with the increase of annealing time and ammonia flow (increase of nitrogen content).

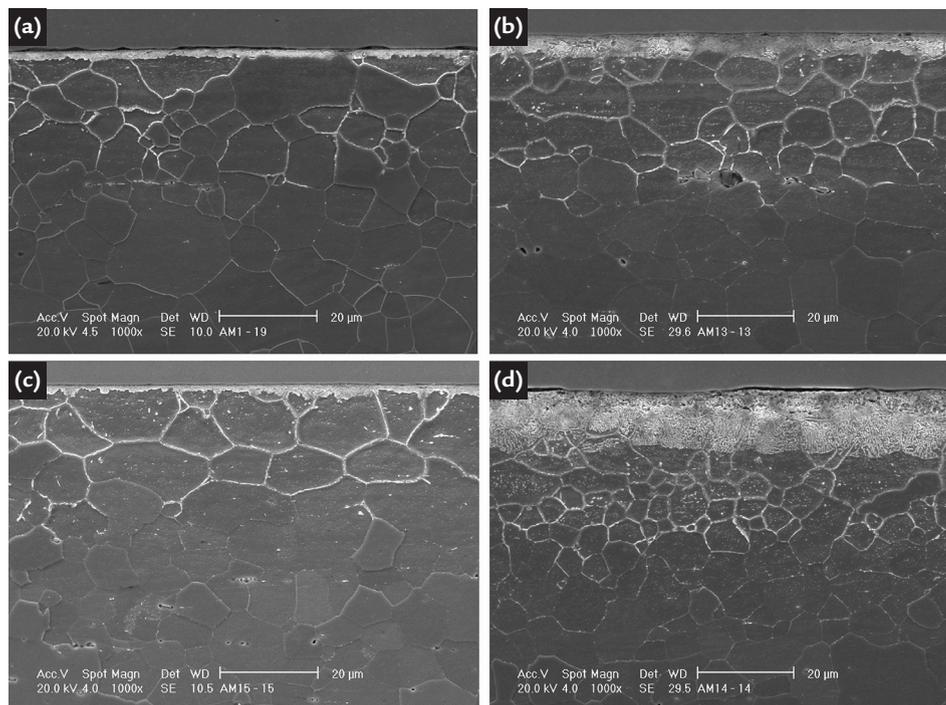


Figure 5 Samples after decarburization and nitriding: a) 63.6s, 0.2Nm<sup>3</sup>/h; b) 119.3s, 0.2Nm<sup>3</sup>/h; c) 63.6s, 0.4Nm<sup>3</sup>/h; and d) 119.25s, 0.4Nm<sup>3</sup>/h. Temperature annealing 860°C. SEM.

Vigorous secondary recrystallization occurred on all the samples with an average final grain size around 5.5mm, very

close to the typical value for GO produced by low slab reheating temperature (Fig. 6). In spite this, crystalline orientation

obtained has around 20° average deviation from Goss orientation. (Fig. 7 and Table 2).

	860°C	900°C
t=63.6s NH <sub>3</sub> = 0.1Nm <sup>3</sup> /h		
t=63.6s NH <sub>3</sub> = 0.2Nm <sup>3</sup> /h		
t=63.6s NH <sub>3</sub> = 0.4Nm <sup>3</sup> /h		
t=95.4s NH <sub>3</sub> = 0.1Nm <sup>3</sup> /h		
t=95.4s NH <sub>3</sub> = 0.2Nm <sup>3</sup> /h		
t=95.4s NH <sub>3</sub> =0.4 Nm <sup>3</sup> /h		
t=119.25s NH <sub>3</sub> = 0.1Nm <sup>3</sup> /h		
t=119.25s NH <sub>3</sub> = 0.2Nm <sup>3</sup> /h		
t=119.25s NH <sub>3</sub> = 0.4Nm <sup>3</sup> /h		

Figure 6 Final grain structures macrographs after box annealing treatment. OM. Samples width = 3cm.

Pole figure	B (mT)	W(W/kg)	N (ppm)	D <sub>p</sub> (µm)	t (s)	NH <sub>3</sub> (N/m <sup>3</sup> )	T (°C)	β
(a)	1713	1.539	533	10.1	63.6	0.1	900	18.9°
(b)	1626	1.906	2324	8.5	119.3	0.2	860	19.6°
(c)	1666	1.739	2885	8.6	119.3	0.4	860	20.6°

Table 2 Characteristics of pole figures: B=magnetic induction; W=core loss; D<sub>p</sub>=primary grain size; β=average deviation of ideal Goss direction.

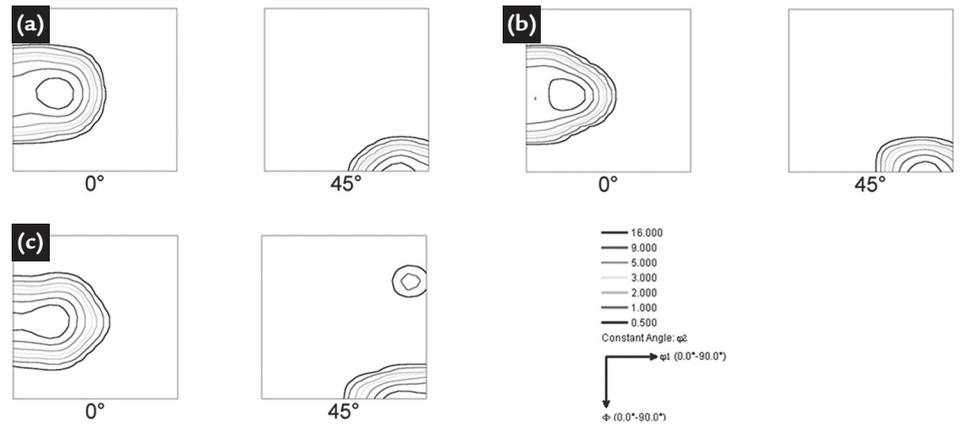


Figure 7  
Average Goss  
orientation deviation for a  
three sample analysed by EBSD:  
a) 900°C, 63.6s, 0.1Nm<sup>3</sup>/h; b) 860°C,  
119.3s,0.2Nm<sup>3</sup>/h; c) 860°C,19.3s,0.4Nm<sup>3</sup>/h.

**Magnetic properties**

Fig. 8 and 9 show the magnetic induction at 800 A/m as a function of the nitrogen content and primary grain size, respectively, at annealing temperatures

860 and 900°C. The magnetic induction of the strip steel decreased with the increase of nitrogen content. There was a decrease in the average primary grain size with the

increase of nitrogen content, which resulted in a decrease of the magnetic induction. Similar behaviour has been observed for magnetic core loss (Fig. 10).

Figure 8  
Magnetic induction  
as a function of nitrogen content  
after decarburization and nitriding.

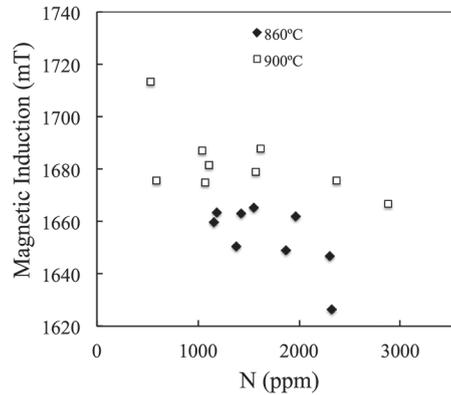


Figure 9  
Magnetic induction  
as a function of primary grain size.

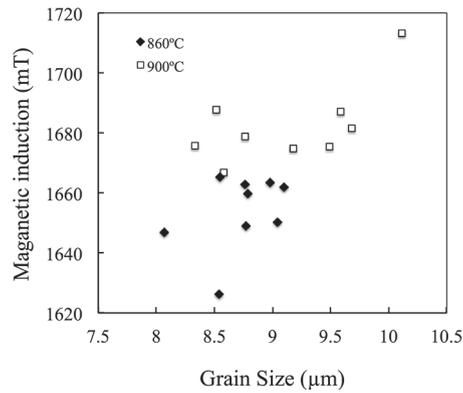
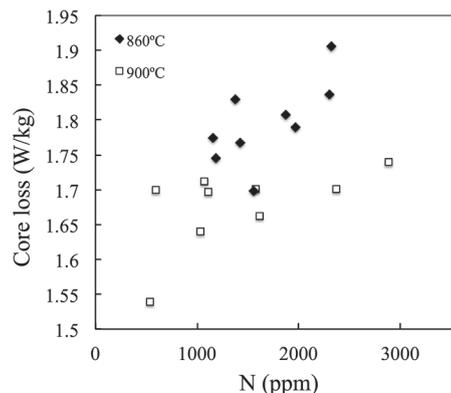


Figure 10  
Magnetic core loss  
as a function of nitrogen content  
after decarburization and nitriding.



The crystalline orientation (Fig. 7) observed in all the samples, with about

20° average deviation from ideal Goss orientation, resulted in values of magnetic

induction (B8) varying from 1.63 to 1.72T, and the magnetic core loss varying from

1.53 to 1.91W/kg, while with standard process, 1.90T and 1.40W/kg can be obtained. This crystallographic texture can be explained by the small primary grain size and the large nitrogen content of the strips after the simultaneous decarburization and nitriding processes. These processes decreased the onset of a secondary recrystallization temperature, allowing the nucleation and growth of grains with

others crystalline orientations. Table 2 presents the characteristics of the three samples tested with small primary grain size and high nitrogen content, resulting in a high average deviation of ideal Goss orientation (Joo *et al.*, 2008).

The possibility of success of this process could be increased, if measures such as increasing primary grain size and reduction of strip nitrogen content

are taken. In this sense, the incoming strip nitrogen content and ammonia flow could be reduced. In addition, primary grain growth inhibition could be reduced by acting in a chemical composition, slab reheating temperature and hot band annealing thermal cycle parameters. These actions could accelerate the grain growth during the simultaneous decarburization and nitriding processes.

#### 4. Conclusions

The increase of ammonia flow increased the nitrogen amount in the samples hindering the primary grain growth and adversely affected the magnetic properties. The increase of annealing

temperature improved the decarburization and the magnetic properties. The best magnetic properties, induction of 1713mT and core loss of 1.54W/kg, were found in the sample with the highest primary grain

size and the lowest amount of absorbed nitrogen. The chance of success of this process could increase if a higher primary grain size and lower nitrogen content added in the strip were achieved.

#### Acknowledgment

The authors would like to thank Aperam South America by using of its

facilities and CAPES for supporting this study.

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Received: 15 May 2017 - Accepted: 5 June 2018.



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