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Original Article

# Secondary recrystallization characteristics of 3%Si grain-oriented electrical steel

*Afonso Vaz de Mello Cardoso*[a], *Sebastião da Costa Paolinelli*[b], *Carolina Cesconetto Silveira*[b], *André Barros Cota*[c,*]

[a] *REDEMAT, Federal University of Ouro Preto, Ouro Preto, MG, Brazil*
[b] *Research Department, Aperam South America, Timóteo, MG, Brazil*
[c] *Department of Physics, Federal University of Ouro Preto, Ouro Preto, MG, Brazil*

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ABSTRACT

A secondary recrystallization Lab study focusing on the secondary recrystallization temperature and magnetic properties was carried out on cold rolling industrial samples of 3%Si grain-oriented electrical steel produced by low slab reheating temperature technology. The results showed a secondary recrystallization temperature, determined by the observed grain size change, of 1080 °C with annealing interruption and of 1060 °C in annealing for 50 h at constant temperature. In addition, the best set of magnetic properties, the higher induction and the lower core loss values, was achieved by annealing the samples during 50 h at 1070 °C constant temperature.


## 1. Introduction

Grain-oriented electrical steel is an important material used as lamination cores in several types of transformers. Its magnetic properties, high magnetic induction and low core loss are closely related to the Goss texture {110}⟨001⟩sharpness obtained by secondary recrystallization. In general, the normal grain growth inhibition by second phase particles is the essential condition for secondary recrystallization [1–4].

In the acquired inhibitor technology, the main inhibitor AlN is obtained by the strip nitriding with cracked $NH_3$ after decarburization annealing [2,6]. These particles take important role during secondary recrystallization annealing making it possible to obtain Goss texture compatible with the high-permeability grain-oriented steels requirements [5–7]. AlN particles, as grain growth inhibitor, and the secondary recrystallization annealing conditions are key parameters to achieve proper final texture and magnetic properties [5,6,8,9].

Some studies have reported the effects of primary grain size and texture on structure evolution during secondary recrystallization [3], as well as of nitriding and heat treatment conditions on secondary recrystallization behaviours and magnetic properties [5,6,9]. The relationship between inhibitors and secondary recrystallization of grain-oriented electrical steel was also reported [10]. Woo et al. [11] have reported the influence of the grain size and nitrogen content

* *Corresponding author.*
E-mail: abcota@ufop.br (A.B. Cota).
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after primary recrystallization on the sharp Goss texture evolution. They have shown that the onset secondary recrystallization temperature determined by the microstructure change is not exactly the same one determined by the change of magnetic induction. They have used the temperature at $B_8 = 1.6$ T (magnetic induction at 800 A/m) as the onset temperature of secondary recrystallization.

In this work, the evolution of the secondary recrystallization was investigated by microstructure and magnetic properties characterization using samples of 3%Si grain-oriented electrical steel industrially processed with low slab reheating temperature technology, collected after decarburizing and nitriding steps.

## 2. Experimental

The steel used was a grain-oriented (GO) containing 3.12Si, 0.044C, 0.15Mn, 0.032Al, 0.0068N, 0.007S (chemical composition in weight %). Samples were industrially processed with low slab reheating temperature – 1150 °C, hot rolled in a Steckel reversible mill to the thickness of 2.3 mm, initially annealed at 1000 °C, cold rolled to 0.27 mm, and then they were nitrided by $NH_3$ atmosphere after the decarburizing annealing at 860 °C.

Primary recrystallized specimens coated with MgO were heated at constant heating rate of 10 °C/h in 75%$H_2$ + 25%$N_2$ atmosphere. During heating, the specimens were extracted at temperatures of 950, 1000, 1050, 1060, 1070, 1080, 1090, 1100, 1150 and 1200 °C (annealing interruption) and cooled down to the room temperature. This model of annealing was used to determine secondary recrystallization temperature ($T_{rcm}$) by checking microstructure change. Other samples coated with MgO were submitted to the box annealing at an interval of 10 °C in the temperature range between ($T_{rcm} - 50$) °C and ($T_{rcm} + 10$) °C for 50 h in an atmosphere of 75% $H_2$ + 25%$N_2$ and cooled down to room temperature. This current production annealing was used to determine the secondary recrystallization temperature by magnetic induction change ($T_{rc}$).

The microstructure of the samples after nitriding annealing was characterized using optical (OM) and scanning electron microscopy (SEM). Chemical analyses of carbon and nitrogen were made using the Leco device. After secondary recrystallization annealing, $B_8$(T) value (magnetic induction at 800 A/m) and $W_{17/60}$ (W/kg) value (magnetic loss at 1.7 T and 60 Hz) were measured in Brockhaus MPG10D device with strips of 100 mm × 30 mm. The secondary grain size was measured manually and chemical analyses were performed to determine the carbon, nitrogen and sulphur after the secondary recrystallization annealing, without interruption.

## 3. Results and discussion

After decarburizing and nitriding annealing, the recrystallized samples showed the mean grain size of 21.6 ± 1.3 μm (Fig. 1) and the amount of carbon and nitrogen was 11.7 ± 0.9 ppm and 253 ± 7 ppm, respectively.

The mean grain size of the samples submitted to secondary recrystallization annealing with interruption as a function of temperature is shown in Fig. 2 Secondary recrystallization temperature was defined as the temperature where the complete secondary recrystallization structure (100% abnormal grains) is observed. AlN precipitates are very effective in preventing normal grain growth and their coarsening and dissolution lead to abnormal grain growth at high temperatures. It can be observed in Fig. 2 that small grains remain up to 1070 °C and at 1080 °C abnormal grain growth occurs, producing a very large grain size. The secondary recrystallization temperature can be determined by mean grain size change, and abnormal grain growth occurred at 1080 °C ($T_{rcm}$).

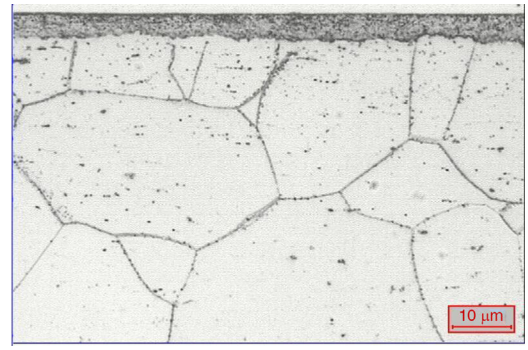


**Fig. 1 – Microstructure of the sample after decarburization and nitriding annealing.**

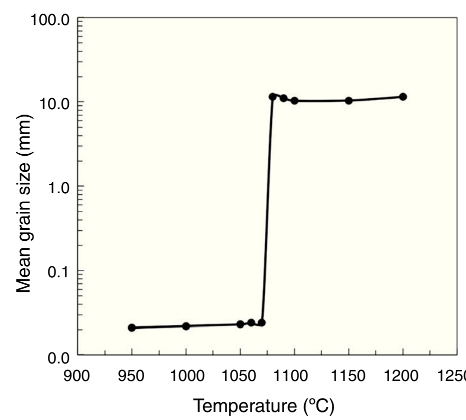


**Fig. 2 – Mean grain size after secondary recrystallization annealing with interruption as a function of the temperature.**

Fig. 3 shows the microstructures of samples annealed at 1070 and 1080 °C and the respective macrostructures are shown in Fig. 4 When the annealing temperature increased from 950 °C to 1070 °C the mean grain size increased from 20.6 ± 1.2 μm to 23.8 ± 1.5 μm. At annealing temperature of 1080 °C, the mean grain size was 11.5 ± 0.9 mm.

The secondary recrystallization temperature determined by grain size change, $T_{rmc}$ = 1080 °C, was used as reference to define the temperatures in the box annealing for 50 h at constant temperature: 1030, 1040, 1050, 1060, 1070, 1080 and 1090 °C.

Figs. 5 and 6 show the magnetic induction ($B_8$) and the magnetic loss ($W_{17/60}$), respectively, as a function of the annealing at constant temperature, i.e., after the secondary

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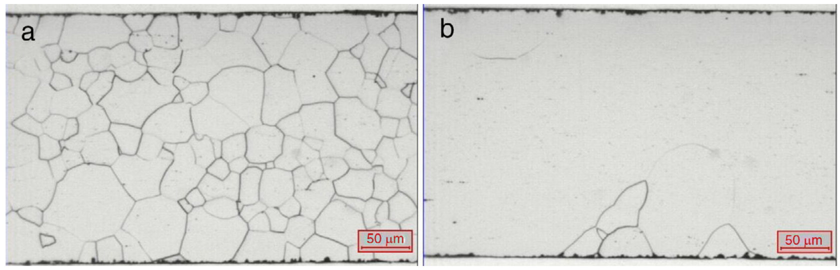


**Fig. 3 – Microstructure of samples annealed at (a) 1070 °C and (b) 1080 °C with interruption.**

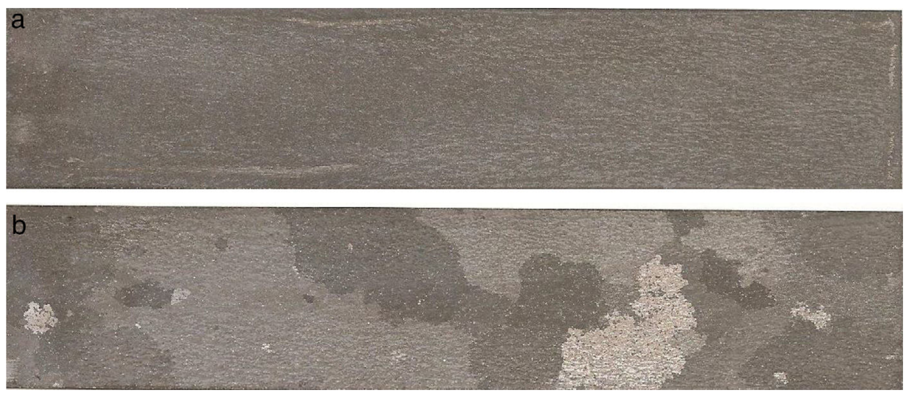


**Fig. 4 – Macrostructure of samples annealed at (a) 1070 °C and (b) 1080 °C with interruption.**

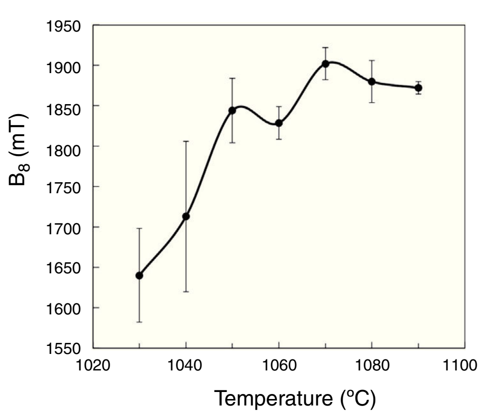


**Fig. 5 – Magnetic induction ($B_8$) change with the annealing at constant temperature.**

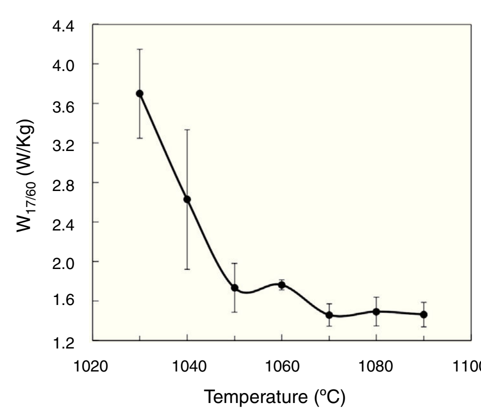


**Fig. 6 – Magnetic loss ($W_{17/60}$) change with the annealing temperature at constant temperature.**

recrystallization annealing for 50 h. Magnetic induction increases up to 1070 °C and achieves a maximum of 1.90 ± 0.02T; the magnetic loss decreases until the temperature value and shows a minimum of 1.46 ± 0.11 W/kg.

Macrostructures of the specimens after secondary recrystallization annealing are shown in Fig. 7 When the annealing temperature increased from 1030 to 1050 °C, a partial secondary recrystallization is observed and the grain size distribution is heterogeneous (Fig. 7a–c). Above 1060 °C, the secondary recrystallization is complete and a more homogeneous grain size distribution is observed (Fig. 7d–g). At 1060 °C the percentage of abnormal grains reached 100% and the secondary recrystallization is completed (Fig. 8) The longer annealing time used in this method resulted in a secondary recrystallization temperature a little lower.

Observing the evolution of magnetic properties with the annealing temperature in Figs. 5 and 8 the best set of magnetic properties is achieved at 1070 °C. This temperature value is between the two temperature recrystallization values pointed out by the two previous methods. This value 1070 °C ($T_{rc}$) corresponds to the maximum magnetic induction ($B_8$) and minimum magnetic loss ($W_{17/60}$). In spite of other influences on magnetic properties beyond those micro structural analyzed, it is reasonable to accept this temperature as a reference for the secondary recrystallization temperature for this steel according to these experiment conditions.

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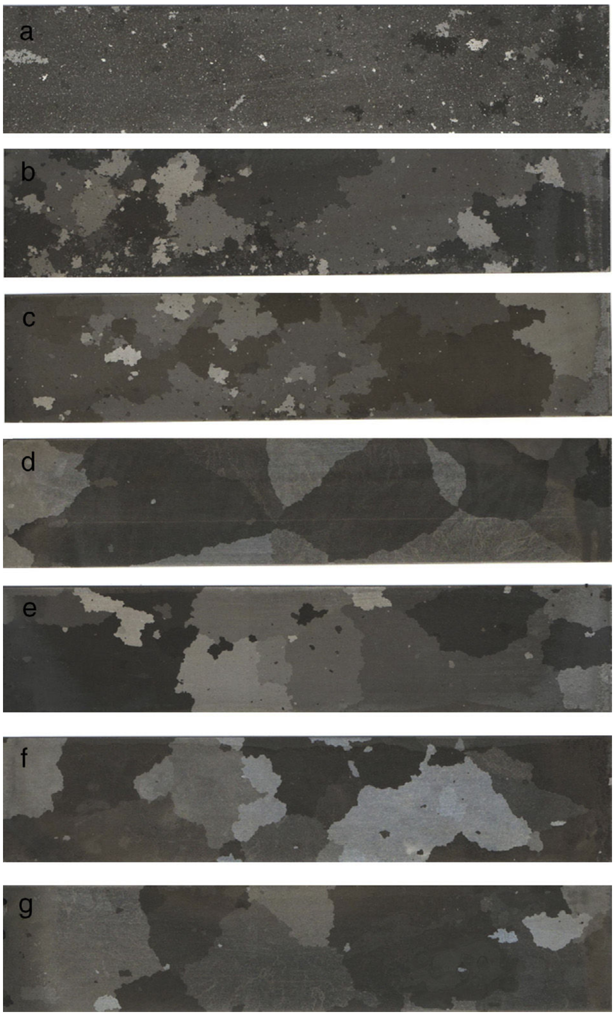


**Fig. 7 – Macrographs of the samples annealed for 50 h at different temperatures: (a) 1030 °C, (b) 1040 °C, (c) 1050 °C, (d) 1060 °C, (e) 1070 °C, (f) 1080 °C, (g) 1090 °C.**

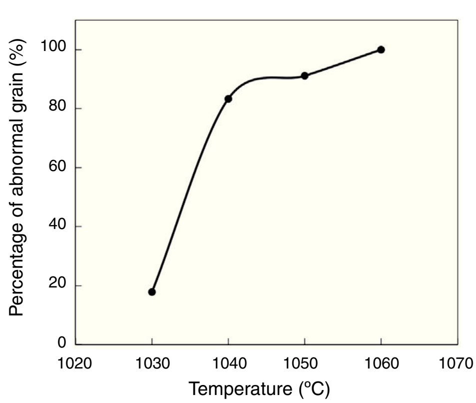


**Fig. 8 – Percentage of abnormal grains as a function of annealing temperature for 50 h.**

The mean grain sizes after secondary recrystallization with annealing at constant temperature kept increasing a little with the annealing temperature and achieved a value of 15.1 mm at 1090 °C. The magnetic loss depends on mean grain size after secondary recrystallization and this effect is just a manifestation of a changing restraint to magnetic domain wall motion. First, grain boundaries constitute a major restraint to domain-wall motion and an increase in grain size decreased the magnetic loss up to temperature of 1070 °C. Above 1070 °C, the increase in the grain size results in an increase in the 180° main domain spacing and in an increase of the magnetic loss apparently because of the eddy currents generated by the moving walls, as was suggested by Pry and Bean [11]. The effect of the purification that occurred at the following temperatures could minimize the effect of grain size increase making the core loss values almost constant after 1070 °C.

After the completion of the annealing, the degree of the deviation of ⟨100⟩ direction of secondary grains from the ideal Goss orientation can be estimated by using the magnetic induction $B_8$, because this value is close to the saturation, where the magnetization depends on the domain rotation that is more dependent on the crystalline orientation. The increase of the magnetic induction with annealing temperature (Fig. 5) up to 1070 °C ($T_{rc}$) is associated to the increase of the percentage of abnormal grains that achieves 100% at 1070 °C, which corresponds to the minimum deviation from the ideal Goss orientation achievable according to these experiment conditions. At secondary recrystallization temperature of 1070 °C, the magnetic induction was 1.90T and the average deviation from the Goss orientation can be estimated about 3–4° [12]. Above 1070 °C, $B_8$ decreases a little, probably as a consequence of a small deterioration on texture due to the appearance of small grains in the structure of the samples.

After secondary recrystallization, the increase of the annealing temperature from 1030 to 1090 °C reduced the carbon content from 12 to 7 ppm, nitrogen content from 119 to 40 ppm and sulphur content from 30 to 10 ppm. When the annealing temperature increases, the dissolution of AlN increases the amount of nitrogen in solid solution that diffuses out of the sheet as gas at high temperature into $H_2$ atmosphere, reducing its content after secondary recrystallization. These phenomena have a positive effect, reducing core loss.

## 4. Conclusions

The secondary recrystallization temperature is a key parameter to optimize the final annealing of grain-oriented electrical steels. This work arrived to the values: at 1080 °C by mean of the annealing with interruption and at 1060 °C annealing for 50 h. The secondary recrystallization temperature determined by magnetic properties change was 1070 °C, which corresponds to the maximum value of magnetic induction ($B_8$ = 1.90 T) and the minimum magnetic loss ($W_{17/60}$=1.46 W/kg). Those methodologies can contribute as a complementary means to define the secondary recrystallization temperature for grain-oriented electrical steels.

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## Conflicts of interest

The authors declare no conflicts of interest.

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