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# Influence of the Annealing Parameters on Core Losses in High-Silicon(6.4 wt.-%) – Iron Electrical Steels Obtained both by Rapid Quenching and by CVD Enrichment

By *Ignacio Ibarrondo** and *Jose Maria San Juan*

The iron and steel production process for manufacturing non-oriented magnetic steel sheets presents several research and development challenges,[1,2] including the production of high-silicon alloys (4–6.5 wt.-%).

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These alloys can be obtained directly from the molten state by rapid quenching or by silicon enrichment of conventional non-oriented magnetic steel sheets (≈3 wt.-% Si) by chemical vapor deposition (CVD).

The material thus obtained can be used in a wide variety of industrial applications, the most important of which is core production for high-performance electrical machines: electrical AC motors (≈400 Hz) in on-board equipment; small (<0.75 Kw) high-speed induction motors working at frequencies of 50–200 Hz; and transformers operating at 200–10 000 Hz frequency range in electronic power supply.

Considering the economic importance of these products, it is evident that the effect of the production process, in particular the heat treatment step, on the final magnetic characteristics of the material, is worthy of study.

This communication describes the study of the influence of the temperature, atmosphere, and heat treatment parameters on the final core losses (0.8–1.0 T, 50–1.000 Hz) for material obtained by rapid solidification and for material obtained by CVD.

Our aim is the optimization of the heat treatment as this is of considerable economic and technical relevance for future manufacturing processes.

In fact, this material, due to its high silicon content (≈6.4 wt.-%), presents extremely low magnetostriction and high resistivity in relation to the conventional (≈3 wt.-%Si) materials.[3] If, after the final heat treatment, we obtain a minimum level of residual tension stresses, along with an adequate grain size (150–200 μm)[4] and at the same time induce a non-oriented cube texture {100}<0*kl*>,[5] which eliminates the hard-to-magnetize <111> directions from the sheet plane, the result is a material especially suitable for the applications previously stated.

In fact, these variables are a function of the parameters involved in the previous manufacturing process and, in particular, of the heat-treatment parameters. In fact, this treatment[6–8] has a decisive influence on grain size and texture, since the type of recrystallization obtained, whether primary or tertiary, is a function of the soaking time, temperature range, and the type of atmosphere used ($N_2$, $H_2$, Ar, vacuum).[9,10] In the case of the material enriched in silicon by CVD, the heat treatment is more complex and consists of two steps: the siliconizing of rolled, conventional silicon steel sheet, using the CVD technique, with a silicon tetrachloride ($SiCl_4$) atmosphere, and back diffusion, homogenizing the silicon content inside the bulk of the steel.

Consequently, to the time and temperature parameters, previously mentioned, it is necessary to add the $SiCl_4$ concentration, gas flow, and gas velocity inside the furnace atmosphere, since all these parameters have a great influence in the different steps of the process:

- Mass transfer ($SiCl_4$ and $FeCl_2$) in the boundary layer, in addition to the adsorption ($SiCl_4$) and desorption ($FeCl_2$) processes on the surface of the material and the kinetics of the surface reaction $SiCl_4 + 5Fe \rightarrow Fe_3Si + 2FeCl_2$.
- Silicon diffusion process inside the material.

In accordance with our objective, microcrystalline ribbons (6.4 wt.-% Si, 25 mm × 38.2 μm) were obtained by rapid quenching,[11] and then submitted to the heat treatment processes described in Table 1. Similarly, non-oriented magnetic steel (≈3.0 wt.-% Si, 20 mm × 50 μm) was siliconized and homogenized according to the process parameters described in Table 1.[12]

For both materials, the micrographic structure, average grain size,[13] and induced texture,[14] were determined. These results are shown in Figure 1 and Table 2.

Likewise, the core losses for each of the heat treated materials described in Table 1 were determined at different inductions (0.8–1.0 T) in the 50–1000 Hz frequency range using the experimental equipment developed by J. Degauque et al.[15]

*Table 1. Annealing cycles.*

| I: Material obtained by rapid solidification | | | | | |
|---|---|---|---|---|---|
| Atmosphere | Gas flow (m/s) | Annealing | | | |
| | | Temp(°C) | Time (min) | | |
| | | | Heating | Soaking | Cooling (40°C) |
| $N_2$, Ar (I) | 0.059 | 900 | 19–22 | 180 | 358–369 |
| $N_2$, Ar (II) | 0.059 | 1100 | 26–29 | 180 | 393–405 |
| Vacuum (1–20 Pa) | – | 1050 | 35–39 | 180 | 450–510 |
| II: Conventional N.O. material silicon enriched by CVD | | | | | |
| Siliconizing step | | | | | |
| $SiCl_4$20%+$N_2$ | 0.017 | 1200 | 30–35 | 15–20 | – |
| Diffusion step | | | | | |
| $N_2$ (99.999%) | 0.059 | 1200 | – | 10–25 | 420–435 |

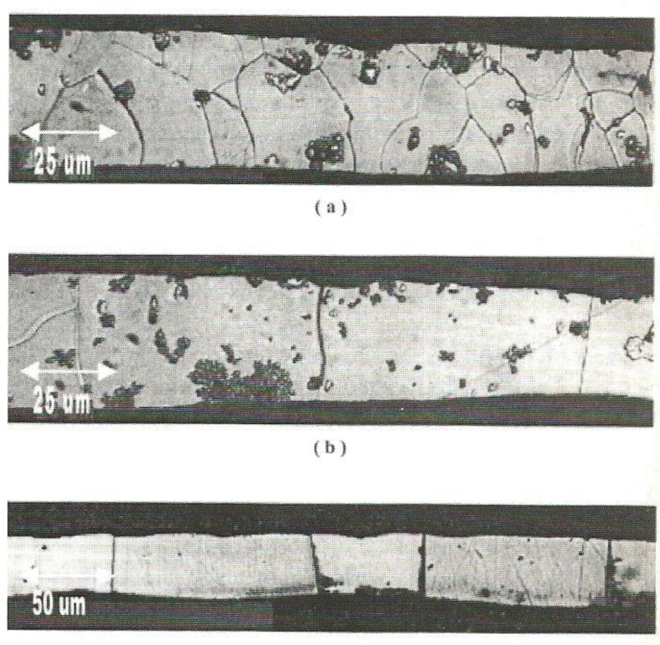


*Fig. 1. Micrographic structures: material annealed under $N_2$/Ar atmospheres. a) Cycle I (900 °C) D < t; b) cycle I (900 °C) D > t; c) Cycle II (1100 °C), D > t. D = average grain size, t = material thickness.*

Figure 2 shows the results obtained at 1.0 T, indicating the results corresponding to the non-oriented conventional magnetic materials.

In view of the results obtained, we have analyzed the influence of the annealing temperature and the type of atmosphere on texture and grain size and, therefore, on the magnetic characteristics of the material.

*Influence of the heat treatment temperature.* Regarding grain size, it can be deduced from the micrographic observations of the samples (Figure 1 and Table 2) that the material heat treated with Cycle I (900 °C), regardless of the atmosphere type, presents two clearly differentiated micrographic structures, depending on whether the average grain size, $D$, is smaller ($D \approx 15.9$ µm) or larger ($D \approx 93.4$ µm) than the material thickness, $t$. This fact suggests that an unfinished recrystallization process has occurred under Cycle I (900 °C), in the sense that residual stresses remain inside the material.

With respect to the influence of temperature on the texture obtained it can be deduced, from the analysis of the data in Table 2, that the complete elimination of the unwanted texture component {222} in relation to the as-quenched state has been achieved, and that the {310}<*hkl*> component remains predominant in all cases, except for the material annealed under vacuum.

*Table 2. Grain size and texture.*

| Atmosphere | Grain Size (µm) | | Texture Area [{*hkl*}/{Total} %] | | | | |
|---|---|---|---|---|---|---|---|
| I: Material obtained by rapid solidification | | | | | | | |
| | $D<t$ | $D>t$ | 110 | 200 | 210 | 310 | 222 |
| As-quenched | 9.1 | – | 37.5 | 4.0 | 13.4 | 33.9 | 10.8 |
| $N_2$, Ar (I) | 15.9 | 93.4 | 13.1 | 0.7 | 4.6 | 81.6 | – |
| $N_2$, Ar (II) | – | 138.4 | 9.6 | 14.1 | 19.1 | 57.1 | – |
| Vacuum (1–20 Pa) | – | 1100 | 12.3 | 76.6 | – | 11.1 | – |
| II: Conventional N-O material silicon enriched by CVD | | | | | | | |
| Siliconizing step $SiCl_4$(20%)+$N_2$ + Diffusion step $N_2$ (99.999 %) | – | 152 | 17.3 | 21.0 | 6.5 | 55.2 | – |

*$D$ = average grain size, $t$ = thickness of the material.*

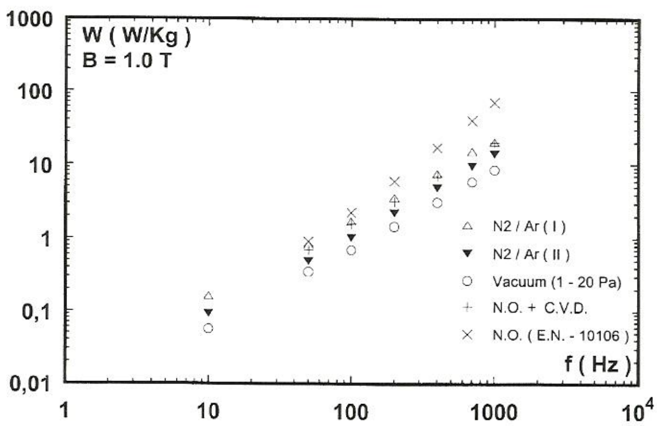


*Fig. 2. Core losses as a function of the frequency for different heat treatments.*

As a consequence, and bearing in mind the results from Figure 2, it can be stated that the 30–40 % decrease in the amount of core losses at 1.0 T in heat treated materials under Cycle II (1100 °C), compared with Cycle I (900 °C), is basically due more to a process of stress relaxation than to the differences in grain size and texture.

*Influence of atmosphere type.* The incidence of the annealing atmosphere is not especially relevant in the case of $N_2$ or Ar atmospheres (Table 2 and Figure 2), but is significant in the case of vacuum atmospheres (1050 °C, 1–20 Pa), where the oxygen adsorbed on the surface of the material promotes a tertiary recrystallization process ($D \approx 1100$ µm) together with a sharp non-oriented {200}<0*kl*> cube texture. This allows a reduction of 20–25 % in core losses to be obtained with respect to atmospheres of $N_2$ or Ar at 1100 °C.

A complete study of this recrystallization process, as a function of the temperature range (950–1250 °C) and vacuum level ($10^{-3}$–30 Pa), and its effect on the final core losses can be seen in our previous research work.[10]

The material obtained by a CVD siliconizing step ($SiCl_4$ (20 %) + $N_2$, 1200 °C) and a diffusion step ($N_2$, 1200 °C) presents similar results in core losses to the material obtained by rapid quenching and then annealing under $N_2$ or Ar atmospheres at 900 °C.

The results obtained allow us to state that the thermally treated material, whether obtained by rapid quenching or by CVD techniques, presents, in all cases, an amount of core losses inferior to that of the highest grade [FeV–240–35 HA, UNE–EN–10106: 1996] of conventional non-oriented magnetic steel sheet. This becomes particularly important at frequencies above 50–60 Hz.

In the case of materials obtained by rapid quenching, the choice between annealing of type I or II must be based on the comparison between the improvement in the amount of total losses (30–40 %), and the higher cost and processing difficulty implied in the annealing temperature increase.

Likewise, materials with a high silicon content (≈6.5 %) obtained by CVD present magnetic characteristics similar to those obtained by means of Cycle I under nitrogen or argon atmospheres.

The best results were obtained with material manufactured by rapid quenching and then annealed under vacuum atmospheres (1050 °C, 1–20 Pa).

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[2] K. I. Arai, K. Ishiyama, *J. Magn. Magn. Mater.* **1994**, *133*, 233.
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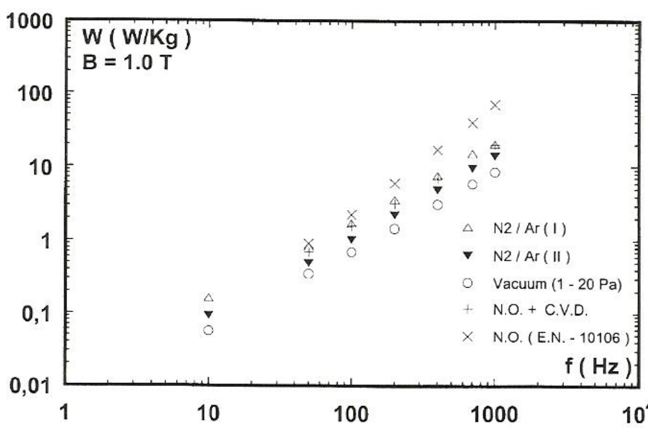


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