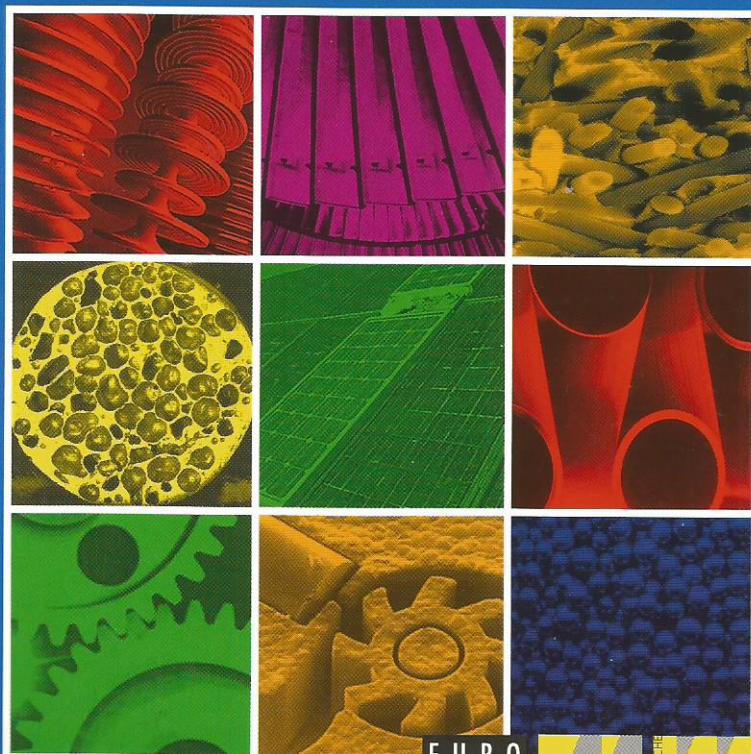


Steels and Materials for Power Plants

EUROMAT – Volume 7

Edited by P. Neumann,
D. Allen and E. Tenckhoff



P. Neumann, D. Allen, E. Teuckhoff (Editors)

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EUROMAT 99 – Volume 7



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Editors:

Prof. Dr. P. Neumann
Max-Planck-Institut für Eisenforschung
Max-Planck-Str. 1
40237 Düsseldorf
Germany

Dr. D. Allen
ABB Asltom Power UK Ltd.
Cambridge Road
Whetstone
Leicester LE9 GLH
United Kingdom

Prof. Dr. E. Teuckhoff
Siemens AG
Postfach 3240
91050 Erlangen
Germany

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Preface

Engineering progress essentially depends on the availability and the intelligent use of materials. For many key industry areas, Europe constitutes a premier place for the development of new materials and their applications. EUROMAT 99, the biannual meeting of the Federation of European Materials Societies with its 21 member societies across Europe set out to become the most comprehensive European event to demonstrate the wide range of the interdisciplinary performance of materials.

EUROMAT was essentially focused on applications of materials with high innovation potential. At the same time, fundamental approaches and processing related aspects for unconventional materials were addressed. In the frame of the 12 conference topics, 1650 papers were originally submitted to the 52 symposia. A total of 655 have been selected as oral presentation by the international group of chairpersons and were presented in 161 sessions. Further, the chairpersons have selected 65 renowned experts for keynote lectures in the frame of their symposium. Roughly 700 papers were displayed as posters.

The scope of EUROMAT was truly international. Papers originated from 57 countries. Among them the major industrial countries of the world have contributed considerably to the wealth of the programme. An overwhelming Eastern European contingent shows that there is a strong interest of these countries in international cooperation.

EUROMAT 99 represents a showcase of the competence of the European materials societies. Various European sister societies and federations act as cosponsors of the event. Joining with FEMS, they are about to establish the network MatNet in order to promote and facilitate their communication and cooperation. They have started a dialogue with the European Commission in order to discuss programme goals and priorities for maintaining Europe's global competitiveness. In view of this promising international perspective, the European Community has agreed to sponsor EUROMAT 99 generously for which we are very grateful. EUROMAT 99 was focused to a large extent on the aims of the closing 4th Framework Programme many projects of which were presented.

EUROMAT 99 was hosted by WERKSTOFFWOCHE, a multisociety joint conference project established in Germany in 1996. Among its initiators is the Deutsche Gesellschaft für Materialkunde, one of the founding member societies of FEMS and technical organiser of this year's EUROMAT.

EUROMAT 99 represented an outstanding success. As the President of FEMS, I would hope that it will serve as a model for future meetings, both in terms of organisation and international cooperation. I would like to extend my gratitude to the scientists, chairpersons and coordinators as well as to the various organisations and particularly to the Messe München who have made this success possible.

Dr. Paul Costa

President of the Federation of European Materials Societies

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Incidence of the Annealing Parameters on Core Losses in High Silicon (6,4% wt) – Iron Electrical Steels Obtained as much by Rapid Quenching as by Enrichment in Silicon by Means of CVD Techniques

I. Ibarrondo , J. M. San Juan

University of Basque Country, San Sebastian, Spain.

1 Abstract

This paper presents the studies carried out on high silicon (6,4% wt) – iron soft magnetic alloys obtained by rapid quenching and by silicon enrichment of conventional non-oriented magnetic steels by C.V.D. techniques.

The influence of the heat treatment parameters : atmosphere type (N_2 , Ar..) and temperature annealing (900 – 1.100 °C) on grain size, texture induced and, therefore, on core losses (1,0 T, 10 – 1.000 Hz.) was studied.

From the analysis of the results it can be deduced that the amount of core losses, is in all cases, lower than that of the highest grade of conventionally processed non-oriented magnetic steel sheet, this being so especially in the range of frequencies above 50/60 Hz.

As a consequence this material is highly adequate for manufacturing cores of high-performance electrical machines working at high frequencies (≈ 400 Hz).

Keywords : Non-oriented magnetic steel sheets, High silicon content (6,4% wt Si), Rapid solidification, C.V.D., Continuous annealing, Magnetic characteristics.

2 Introduction

Nowadays, the siderurgical process for manufacturing non-oriented magnetic steel sheets presents several research and development topic areas (1, 2), amongst which we may highlight the production of high silicon content alloys (4 – 6,5% wt).

These alloys can equally be obtained directly from the melted state by rapid quenching or by enrichment in silicon of conventional non-oriented magnetic steel sheets ($\approx 3\%$ wt Si) by means of Chemical Vapour Deposition (C. V. D.) techniques.

The material obtained in this way can be used in a wide variety of industrial applications, the most important of which is the cores production for high-performance electrical machines:

- Electrical A.C. motors (≈ 400 Hz) in on-board equipments.
- Small ($< 0,75$ Kw) high speed induction motors working at frequencies between 50 – 200 Hz.
- Transformers operating at frequency range in electronic power supply (200 – 10.000 Hz).

Considering the economic importance of the subject in hand, it is evident that the incidence of the siderurgical process – in particular the heat treatment step - on the final magnetic characteristics of the material is worthy of study.

Therefore, the objective of the present paper is the study of the incidence of the heat treatment parameters, temperature and atmosphere type, on the final core losses (0,8 – 1,0 T, 50 – 1.000 Hz) as much for material obtained by rapid solidification as for material obtained by C.V.D. techniques.

The aim of all these procedures is the optimization of the heat treatment as this is of considerable economic and technical significance for future manufacturing processes.

3 Physical Background

In fact, this material, due to its high silicon content ($\approx 6,4\%$ wt), presents extremely low magnetostriction and high resistivity in relation to the conventional ($\approx 3\%$ wt Si) materials (3).

In addition, 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 $\{100\} \langle 0kl \rangle$ (5) which eliminates the directions $\langle 111 \rangle$, hard to magnetize, from the sheet plane, the result is a material especially adequate for the applications previously stated.

These variables, in turn, are a function of the parameters involved in the previous manufacturing process and, particularly, 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 C.V.D., the heat treatment is more complex and consists of two steps: the siliconizing of rolled, conventional silicon steel sheet, using the C.V.D. technique, with a silicon tetrachloride (SiCl_4) atmosphere, and the posterior diffusion, homogenizing the silicon content inside the bulk of 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 incidence 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.



- Silicon diffusion process inside the material.

4 Experimental

In accordance with the objective of the present paper microcrystalline ribbons (6,4% wt Si, 25 mm x 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 ($\approx 3,0\%$ wt Si, 20 mm x 50 μm) was siliconized and homogenized according to the process parameters described in Table 1 (12).

Table 1. Annealing cycles

I.- Material obtained by rapid solidification					
Atmosphere	Gas flow (m/s)	Annealing			
		Temp(°C)	Time (minutes)		
			Heating	Soaking	Cooling (40 °C)
N ₂ , Ar (I)	0,059	900	19 – 22	180	358 – 369
N ₂ , Ar (II)	0,059	1.100	26 – 29	180	393 – 405
Vacuum (1 – 20 Pa)	–	1.050	35 - 39	180	450 - 510
II.- Conventional N.O. material silicon enriched by C.V.D.					
Siliconizing step					
SiCl ₄ 20%+N ₂	0,017	1.200	30 - 35	15 - 20	–
Diffusion step					
N ₂ (99,999%)	0,059	1.200	–	10 - 25	420 - 435

Table 2. Grain size and Texture

I.- Material obtained by rapid solidification							
Atmosphere	Grain Size (μm)		Texture				
	D < t	D > t	Area [{hkl}] / {Total} %				
			110	200	210	310	222
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)	–	1.100	12,3	76,6	–	11,1	–
II.- Conventional N.O. material silicon enriched by C.V.D.							
1° Siliconizing SiCl ₄ (20%)+N ₂ +							
2° Diffusion N ₂ (99,999 %)	–	152	17,3	21,0	6,5	55,2	–

D : Average grain size, t : thickness of the material

For both materials, the micrographic structure, average grain size (13), and texture induced (14), were determined. These results are shown in 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 – 1.000 Hz frequency range using the experimental equipment developed by J. Degauque et al. (15).

Figure 1 shows the results obtained at 1,0 T, indicating the results corresponding to the non-oriented conventional magnetic materials.

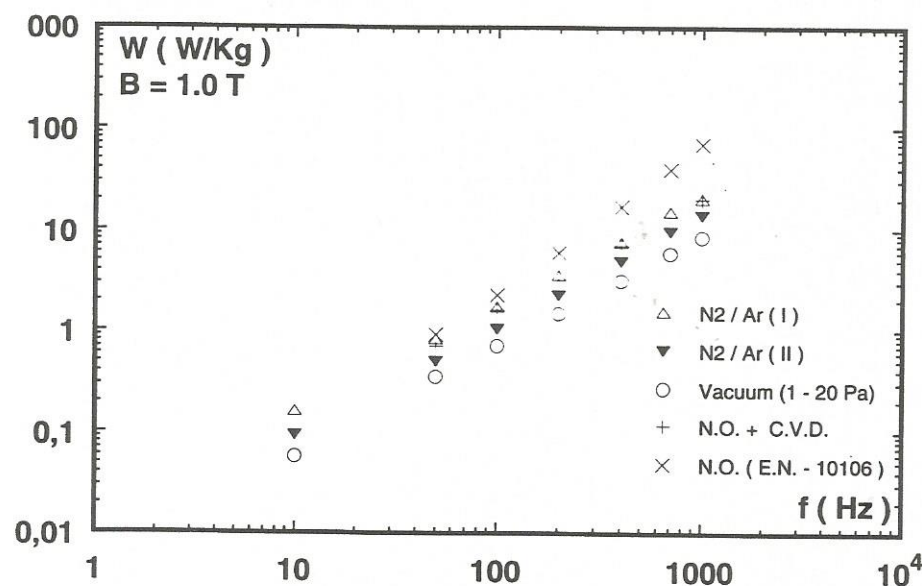


Figure 1. Core losses, as a function of the frequency, for different heat treatments.

5 Results and their Analysis

In view of the results obtained, 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 was analysed.

5.1 Material obtained by rapid quenching

- Influence of the heat treatment temperature on texture, grain size and core losses.

The annealing temperature has little incidence in the texture of the material, with the $\{310\} \langle hkl \rangle$ component remaining as predominant in all cases, except for the material annealed under vacuum.

On the other hand, its incidence in the state of internal stresses inside the material is greater. Therefore the material heat treated under cycle I (900 °C), regardless of the atmosphere type, N₂ or Ar, presents two clearly differentiated micrographic structures, depending on whether the average grain size (D) is smaller or larger 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.

As consequence and bearing in mind the results from figure 1, it can be stated that the 30 to 40 % decrease in the amount of core losses at 1,0 T in heat treated materials under cycle II (1.100 °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 on texture, grain size and core losses.

The incidence of the annealing atmosphere is not so important in the case of N₂ or Ar atmospheres, but is especially significant in the case of vacuum atmospheres (1.050 °C, 1 - 20 Pa), where the oxygen adsorbed on the surface of the material promotes a tertiary

recrystallization process ($D \approx 1.100 \mu\text{m}$) together with a non-oriented $\{200\} \langle 0kl \rangle$ cube texture.

This allows the obtention of a reduction of 20 – 25 % in core losses with respect to the atmospheres of N_2 or Ar at 1.100°C .

5.2 Material obtained by means of C.V.D techniques

The material obtained by C.V.D : siliconizing step (SiCl_4 (20 %) + N_2 , 1.200°C) and diffusion step (N_2 , 1.200°C) presents similar results in core losses to the material obtained by rapid quenching and annealed under N_2 or Ar atmospheres at 900°C .

6 Conclusions

The results obtained in the present paper allow us to state that the thermically treated material, whether obtained by rapid quenching or by C.V.D 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.

The best results were obtained with material manufactured by rapid quenching and annealed under vacuum atmospheres (1.050°C , 1- 20 Pa).

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