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**Metallurgical Processing of High Silicon (6.4%)
Non-Oriented Magnetic Steel Sheets Directly
from the Melted State by Rapid Quenching. A
Study of the Continous Annealing Step.**

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ABSTRACT

This paper presents the studies carried out on microcrystalline ribbons Fe – 6.4%Si previously obtained by rapid quenching. The influence of the atmosphere type (N₂ , H₂ , Ar) and 900 °C (1652 °F) or 1100 °C (2012 °F) temperature range on grain size and texture induced in the material was studied. Likewise, the amount of total core losses in the 10 to 1000 Hz frequency range was measured and the influence of the above mentioned variables on the losses was analysed. 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 200 Hz. Finally, a comparison is made with materials obtained by the same procedure and then annealed under vacuum atmospheres and with those obtained by C.V.D.

1. INTRODUCTION

In the last decade, due to the difficulty of processing these materials by conventional metallurgical processes, several projects have been undertaken in order to obtain microcrystalline ribbons and strips of high silicon level (6.5%) by means of rapid quenching. These new materials are used in the manufacture of cores of high performance electrical machines which work at high frequencies ¹⁻².

It is in this frequency range that this material presents

a considerably lower amount of total core losses in comparison with conventional materials, due to its high (6.5%) silicon content ³ and low gauge ⁴, in addition to the possibility of obtaining the adequate grain size, together with a non – oriented { 100 } < 0kl > cube texture under certain conditions ⁵.

These variables, in turn, are a function of the parameters involved in the previous process of rapid solidification ⁶ and , particularly, of the last heat treatment.

In fact, this treatment has a decisive influence on grain size and texture, since the type of recrystallization obtained, whether primary or tertiary, is a function of the temperature range and the type of atmosphere used.

For this reason, numerous studies have been carried out under different temperatures and various atmospheric vacuum levels ⁷⁻⁹ in order to determine the optimum parameters for this treatment. However, such vacuum atmospheres are difficult and costly to put into industrial practice.

Taking these observations into account, the objective of this project is to optimise the amount of core losses at the lowest possible cost, by using different industrial atmospheres (N₂, H₂, Ar) under two annealing cycles : cycle I at low [900 °C (1652 °F)] and cycle II at high [1100 °C (2012 °F)] temperature respectively. All these studies were carried out in conditions simulating the future manufacturing process.

2. EXPERIMENTAL

In accordance with this objective, [Fe – 6.4% Si, 25mm x 38.2µm (0.98in x 1.50µin)] microcrystalline ribbons were obtained by applying the previously described ¹⁰ production process parameters . The material thus obtained was submitted to the heat treatment processes described in Table I.

For this material, the micrographic structure, average grain size ¹¹, and texture induced ¹², were determined in both the as – quenched state and the heat treated conditions. These results are shown in Table II.

Likewise, the core losses as a function of the temperature (cycles I and II) and of the different annealing atmospheres (N₂, H₂, Ar) were determined at different

Table I: Annealing cycles

Cycle	Atmosphere	Gas Flow (m/s)	Annealing			
			Temp. (°C)	Time (minutes)		
I	N₂, H₂, Ar	0.059	900	Heating 20 – 21	Soaking 180	Cooling (40°C) 362 - 369
II	N₂, H₂, Ar	0.059	1100	27 - 29	180	397 - 409

[(1 m/s. = 39.37 in/s.), (1 °F = 1.8 (°C) + 32)]

Table II: Grain Size and Texture

Amosphere (Cycle)	Grain Size (μm)		Area [{hkl}/{total}] %				
	D < t	D > t	110	200	211	310	222
As - quenched	9.1 (0.23 x t)	---	37.5	4.0	13.4	33.9	10.8
Nitrogen (I)	15.9 (0.41 x t)	93.9 (2.45 x t)	13.1	0.7	4.6	81.6	0.0
Nitrogen (II)	30.0 (0.78 x t)	138.4 (3.62 x t)	9.6	14.1	19.1	57.1	0.0
Argon (I)	15.5 (0.40 x t)	84.9 (2.22 x t)	8.0	0.4	5.4	86.2	0.0
Argon (II)	---	85.6 (2.24 x t)	4.2	10.0	26.8	58.5	0.0
Hydrogen (I)	28.3 (0.74 x t)	174.5 (4.56 x t)	15.6	5.8	3.9	74.6	0.0
Hydrogen (II)	---	189.4 (4.95 x t)	11.0	24.1	5.5	59.2	0.0

(**D** : Average grain size, **t** : thickness of the material , $1\mu\text{m} = 39.37\mu\text{in}$)

inductions (0.8 – 1.0 T) in the 10 – 1000 Hz frequency range using the experimental equipment developed by J. Degauque et al.¹³. Figures 1 and 2 show some of the results obtained at 1.0 T.

3. 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.

3.1 Influence of Heat Treatment Temperature on Texture, Grain Size and Core Losses.

As regards grain size, it can be deduced from the micrographic observations of the samples (Table II) that the material heat treated under cycle I [900 °C (1652 °F)] conditions, regardless of the atmosphere type (N₂, H₂, Ar), presents two clearly differentiated micrographic structures, depending on whether the average grain size (**D**) is smaller than the thickness (**t**) [**D** $\approx$ 15 – 30 μm

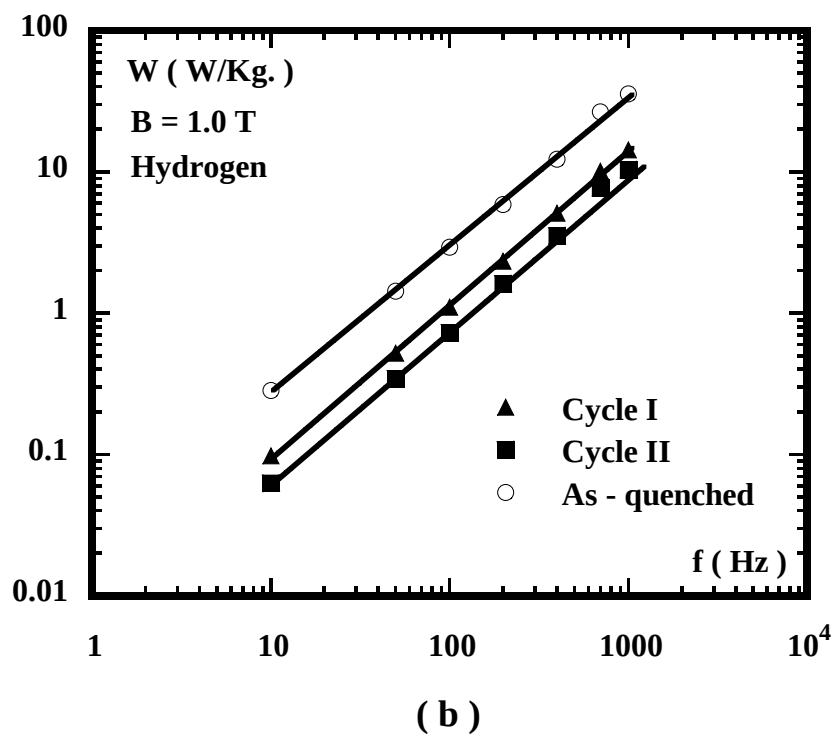
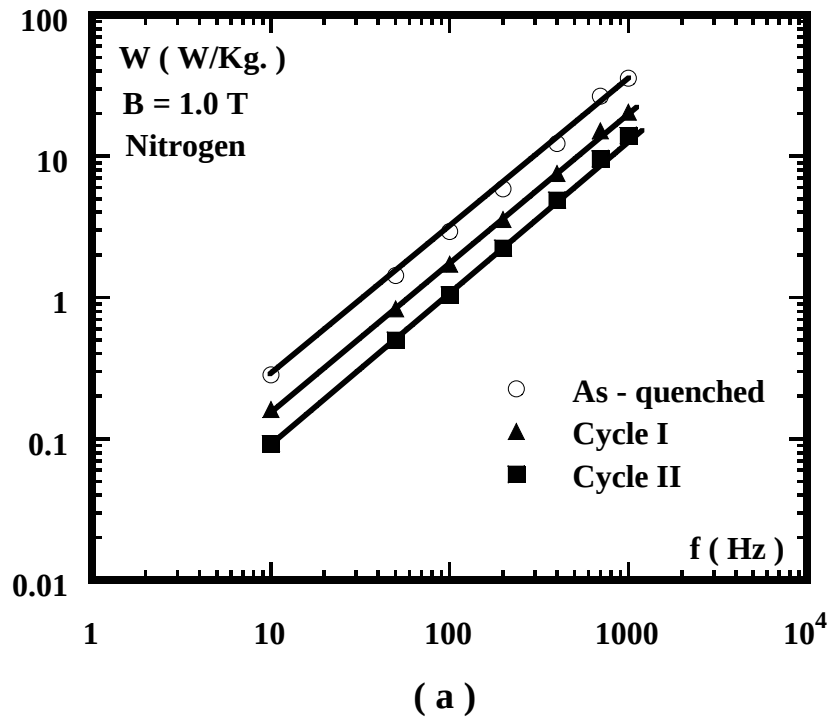


Fig. 1 : Influence of temperature on the amount of core losses
 (1 W/kg = 0.454 W/lb)

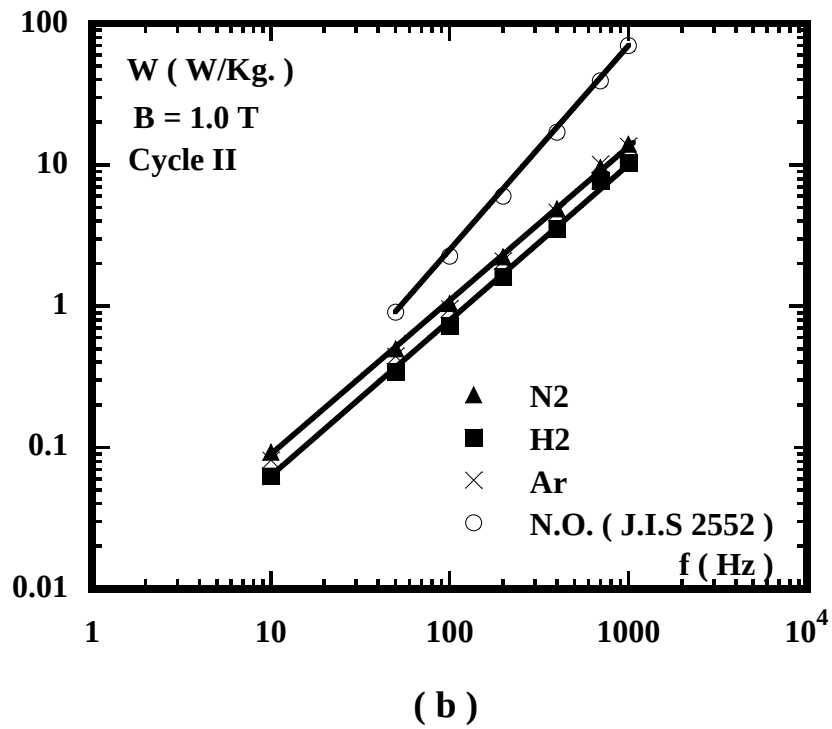
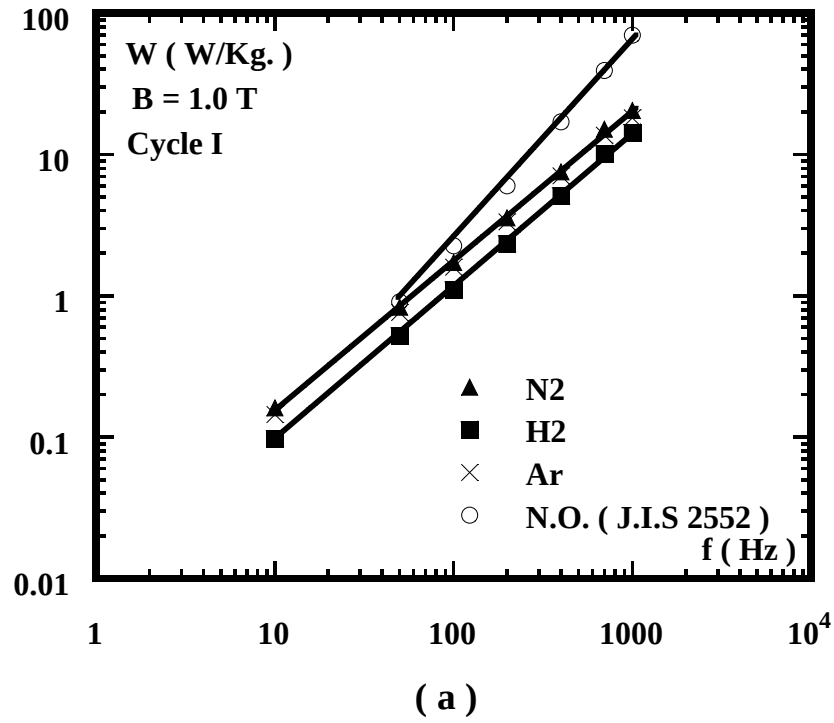


Fig 2 : Influence of atmosphere type on the amount of core losses
(1 W/kg = 0.454 W/lb)

(590.5 – 1181.1 μm) (0.40 – 0.78) $\times t$] or larger [$D \approx 84 - 189 \mu\text{m}$ (3307.1 – 7440.9 μm) (2.22 – 4.95) $\times t$]. This fact suggests that an unfinished recrystallization process has occurred under cycle I, in the sense that residual stresses remain inside the material.

With respect to the influence of temperature on the texture obtained ¹⁴ it can be deduced, from the analysis of the data in Table II, that the complete elimination of the unwanted texture component { 222 } in relation to the as – quenched state has been achieved, and that the component { 310 } < uvw > is predominant in all cases.

The conditions of cycle II, especially in the case of hydrogen, strengthens the { 200 } component of texture, which is particularly important since it eliminates the < 111 > directions which are hard to magnetize from the sheet plane.

Cycle I, however, presents a better texture as regards the positive component { 110 } and the negative one { 211 }.

Figure 1 (a, b) shows the core losses at 1.0 T in the frequency range between 10 and 1000 Hz under nitrogen and hydrogen atmospheres. Similar tests were carried out under argon atmospheres at 1.0 T, and (N₂, H₂, Ar) atmospheres at 0.8 T.

In view of all these results, it can be stated that the 30 to 40 % decrease in the amount of core losses at 0.8 – 1.0 T inductions in heat treated materials under cycle II, compared with cycle I, is basically due more to a process of stress relaxation than to the differences in grain size and texture.

3.2 Influence of Atmosphere Type on Texture, Grain Size and Core Losses.

The influence of the type of atmosphere on grain size and texture is considerably lower than that of the temperature in the case of nitrogen and argon atmospheres.

Thus, when annealing is carried out in these atmospheres (N₂, Ar), the difference in losses between each of the atmospheres, at 0.8 – 1.0 T inductions, varies between 5 – 10 % as can be seen in Figures 2 (a, b) which correspond to cycles I and II respectively.

On the contrary, in the case of hydrogen, there is an improvement in the { 200 } and { 110 } texture components, together with a notable increase in grain size [$D \approx 4.95 \times t$ (maximum $\approx 12 \times t$)] in comparison with the material annealed under nitrogen or argon atmospheres.

This would explain a reduction in losses under a hydrogen atmosphere, in relation to the nitrogen and argon atmospheres, particularly in the case of cycle II (20 – 25%).

4. CONCLUSIONS

The results obtained in the present paper allow us to state that material obtained and treated as explained above presents, in all cases, an amount of core losses inferior to that of the highest grade of conventional non – oriented magnetic steel sheet (H2 – J.I.S. 2552). This, due to the high silicon content of the material, becomes particularly important at frequencies above 200 Hz.

However, it must be pointed out that these results are worse than those obtained when the material is annealed under vacuum¹⁵ where, by means of a tertiary recrystallization process, grain size larger than 1000 μm (3937.0 μm) and a strong non – oriented cube texture can be obtained. Nevertheless, this type of annealing is expensive and difficult to carry out industrially.

Likewise, materials with high silicon content (6.5 %) obtained by C.V.D ¹⁶ present magnetic characteristics similar to those obtained by means of cycle I under nitrogen or argon atmospheres.

In view of these results, for reason of economy and ease of handling, the use of nitrogen atmospheres is recommended for the final heat treatment of these materials.

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.

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Keywords : High Silicon Content (6.4%) Non-Oriented Magnetic Steel Sheets, Rapid Solidification, Continuous Annealing Step, Magnetic Characteristics.

REFERENCES

- York, NY, 1986, pp 46 – 55.
1. A. J. Moses, "Development of Alternative Magnetic Core Materials and Incentives for their Use." J. Magn. Magn. Mater., 112, 1992, pp 150 –155.
 2. F. Fiorillo, "Advances in Fe – Si Properties and their Interpretation." J. Magn. Magn. Mater., 157 / 158, 1996, pp 428 – 431.
 3. K. I. Arai, N. Tsuya, "Ribbon - Form Silicon – Iron Containig Around 6.5 Percent Silicon." I.E.E.E. Trans.Magn., MAG – 16, 1 , 1980, 126 – 129.
 4. M. Pott – Langemeyer, W. Riehemann, W. Heye, "Effect of Thickness on Texture and Magnetic Properties of Meltspun Fe – 6.5 wt % Si Ribbons." Mater. Sci. Eng., A133, 1991, pp 204 – 208.
 5. K. I. Arai, N. Tsuya, K. Ohmori, "Annealing of Silicon – Iron Ribbons Containing Around 6.5 wt % Silicon." I.E.E.E. Trans. Magn., MAG – 17, 6, 1981, pp 3154 – 3156.
 6. Y. Sato, T. Sato, Y. Okazaki, "Production and Properties of Melt – Spun Fe – 6.5 % Si Ribbons." Mater. Sci. Eng., 99, 1988, pp 73- 76.
 7. K. I. Arai, H. Tsutsumitake, K. Ohmori, "Grain Growth and Texture Formation by Annealing of Rapidly Quenched High Silicon – Iron alloys." Trans. Jap. Inst. Met., 25, 12, 1984, pp 855 – 862.
 8. K. I. Arai, K. Ohmori, "Grain Growth Characteristics and Magnetic Properties of Rapidly Quenched Silicon Steel Ribbons." Metall. Trans., 17A, 1986, pp 1295 – 1299.
 9. I. Ibarondo, J. San Juan, J. Gonzalez, "Study of the Incidence of the Last Annealing on the Magnetic Characteristics of High – Silicon (6.0 – 6.5 %) Crystalline Ribbons Directly Obtained from the Melted State by Rapid Quenching." J. Magn. Magn. Mater., 101, 1991, pp 83 – 85.
 10. I. Ibarondo, S. Surinach, J. Gonzalez, "Influence of Manufacturing Process Parameters on Magnetic Characteristics of High – FeSi Crystalline Rapidly Quenched Ribbons." J. Magn. Magn. Mater., 112, 1992, pp 232 – 234.
 11. J. C. Russ, Practical Stereologie , Plenum Press, New – York, NY, 1986, pp 46 – 55.
 12. R.M.S.B. Horta, W .T. Roberts, D. V. Wilson, "Texture Representation by Inverse Pole Figures." Trans. A.I.M.E., 245, 1969, pp 2525 – 2527.
 13. B. Viala, J Degauque, "Proprietes Mechaniques et Magnetiques du Ruban Fer – Silicium 6.5 % Elabore par Solidification Rapide sous Atmosphere Controlee." Thèse Doctoral, Institut National des Sciences Appliquées (I.N.S.A – C.N.R.S), Toulouse, 1994, pp 196 – 198.
 14. H Shimanaka et al., "Recent Development of Non – Oriented Electrical Steel Sheets." J. Magn. Magn. Mater., 26 (1 - 3), 1982, pp 57 – 64.
 15. M. A. Cunha, G. W. Johnson, "Rapidly Solidified Fe – Si Alloys." J. Mat. Sci., 25, 1990, pp 2481 – 2486.
 16. H. Haiji et. al., "Magnetic Properties and Workability of 6.5 % Si Steel Sheet." J. Magn. Magn. Mater., 160, 1996, pp 109 – 114.

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