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Volume XXXVI



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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 Continuous Annealing Steep.**

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October 25 – 28 , 1998**

ABSTRACT

This paper presents the studies carried out on microcrystalline ribbons Fe – 6.4 wt%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 : Vacuum and C.V.D.

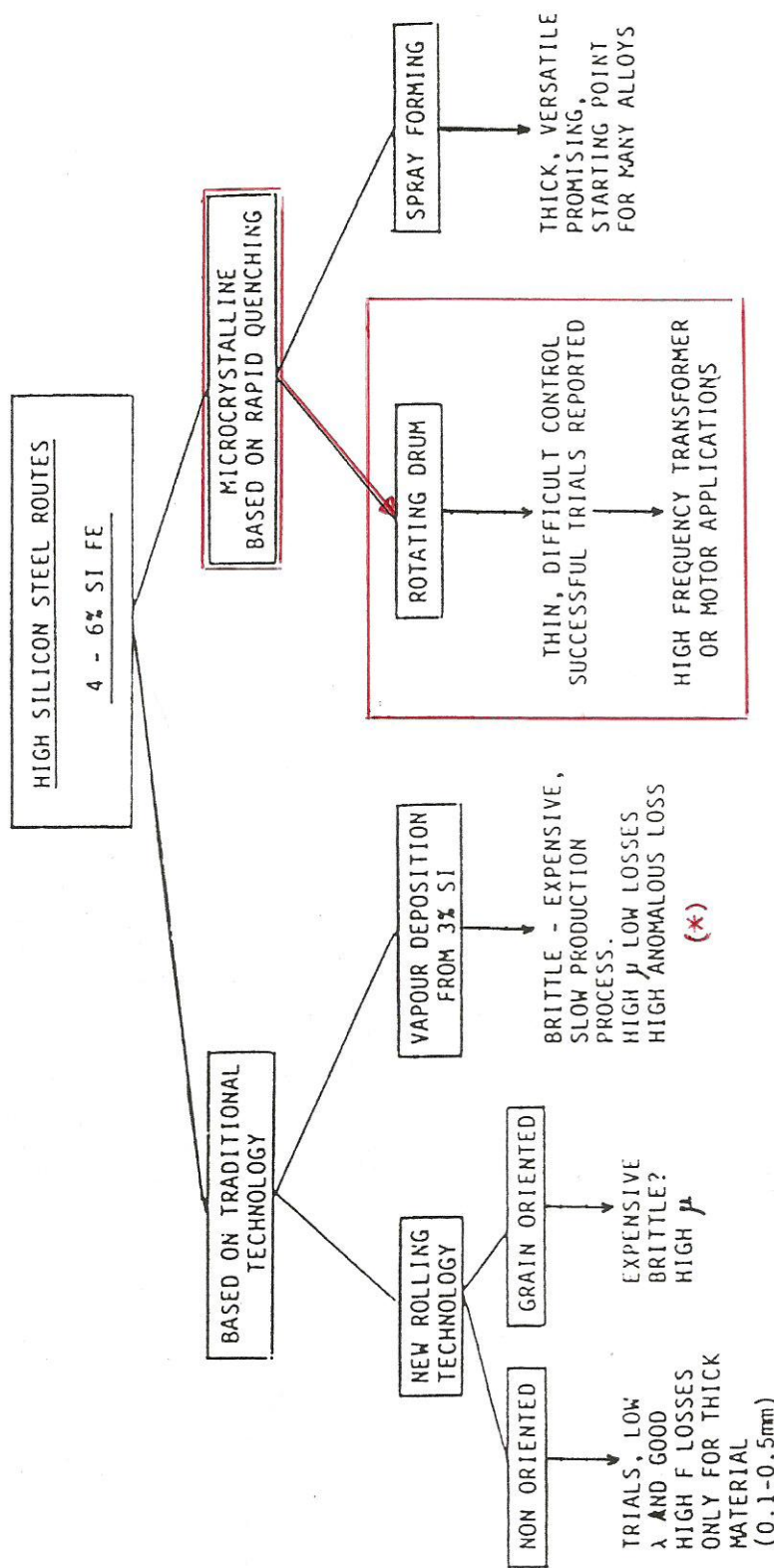


Fig.1. Routes for production of high silicon alloys.

FABRICACION COMERCIAL : N. K. K. Corporation, Japon

N. K. Super E - core ([http : // www. nkk. co. jp](http://www.nkk.co.jp))

Proceso Convencional ($\approx 3\% \text{ Si}$) + C. V. D ($\approx 6.5\% \text{ Si}$)

(1) A. J. Moses, J. Magn., Mater., 112, 1992, 150 - 155.

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- 1.- Introduction. Objectives.
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- 8.- New siderurgical process.

1.- INTRODUCTION

➔ In the last decade, due to the difficulty of processing these materials [electrical steels with high silicon level (≈ 6.5 wt% Si)] by conventional metallurgical processes, several projects have been undertaken in order to obtain microcrystalline ribbons and strips of these materials by rapid quenching.

1.1 – IMPORTANCE OF THIS NEW METALLURGICAL PROCESS.

- * Great simplification of the production process
- * Economy saving : Inversion, maintenance, reduction in labour costs.
- * Energy saving in the production process.

1.2 – IMPORTANCE OF THESE NEW MAGNETIC MATERIALS.

- * Market volume.
- * Main use: Cores production for high performance electrical machines working at (50 - 400 Hz.).

OBJECTIVE

⊗ The objective of the of this research work is to optimise the amount of core losses (50 – 1000 Hz.) at the lowest possible cost, by using :

➔ Different industrial atmospheres (N_2 , H_2 , Ar...)

➔ Under two annealing cycles:

* Cycle I : Low temperature 900 °C,(1652 °F)

* Cycle II : High temperature 1100°C, (2012 °F)

All these studies were carried out in conditions simulating the future manufacturing process.

PHYSICAL BACKGROUND

VARIABLES



* Chemical composition

- Silicon level
- Impurities level (P, S, ..C, N.)

* Thickness of the material

PARAMETERS



RAPID SOLIDIFICATION PROCESS



- * Internal stresses
- * Grain size
- * Texture
- * Internal structure (order - disorder)



HEAT TREATMENT

- Annealing temperature
- Soaking Time
- Atmosphere (N₂, H₂, Ar, Vac.)

THE FINAL MAGNETIC
CHARACTERISTICS
AS A FUNCTION OF

3 - PRODUCTION PROCESS

3.1 – RAPID SOLIDIFICATION

SINTERING

HEATING STEP

Atm. : H₂ (100 %)

Time : 20 – 30 min.

SOAKING STEP

Atm. : H₂ (100 %)

Time : 4 hours

Temp. : 700 °C (1292 °F)

COOLING STEP

Atm. : Argon

MELTING - TAPPING

Melting Temperature : 1550 °C (2822 °F)

Tapping Temperature : 1490 °C (2714 °F)

Injection Pressure : 0.5 atm. (7.34 lb / in²)

ROLLER

Material : Stainless Steel

Diametre : 30 cm (11.8 in)

Angular Velocity : 1800 r.p.m.

 In this way [Fe – 6.4 % Si, 25 mm x 38.2 μm
(0.98 in x 1.5 μin)] microcrystalline ribbons were obtained .

 The material thus obtained was submitted to the
heat treatment processes described in Table I.

3.2 – HEAT TREATMENT

Table I: Annealing cycles

Cycle	Atmosphere	Gas Flow (m/s)	Annealing			
			Temp. (°C)	Time (minutes)		
I	N ₂ , H ₂ , Ar	0.059	900	Heating 20 – 21	Soaking 180	Cooling (40°C) 362 - 369
II	N ₂ , H ₂ , Ar	0.059	1100	27 - 29	180	397 - 409

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

4. – EXPERIMENTAL

I - MICROGRAPHIC STRUCTURE AND AVERAGE GRAIN SIZE:

- * EQUIPMENT : Optical microscopy
- * METHODOLOGY : As described in (1)
- * RESULTS OBTAINED : Figure D and Table II.

(1) J. C. Russ, Practical Stereologie, Plenum Press, New – York, Chap. 3, 1986, pp 46 – 55.

II – TEXTURE INDUCED:

- * EQUIPMENT : X – Ray diffractometer with goniometer
- * METHODOLOGY : As described in (2)
- * RESULTS OBTAINED : Table II.

(2) R.S.M.B. Horta et al. , Trans of Metall. Soc. of A.I.M.E., vol 245, 1969, pp 2525 – 2527.

III. – MAGNETIC CHARACTERISTICS:

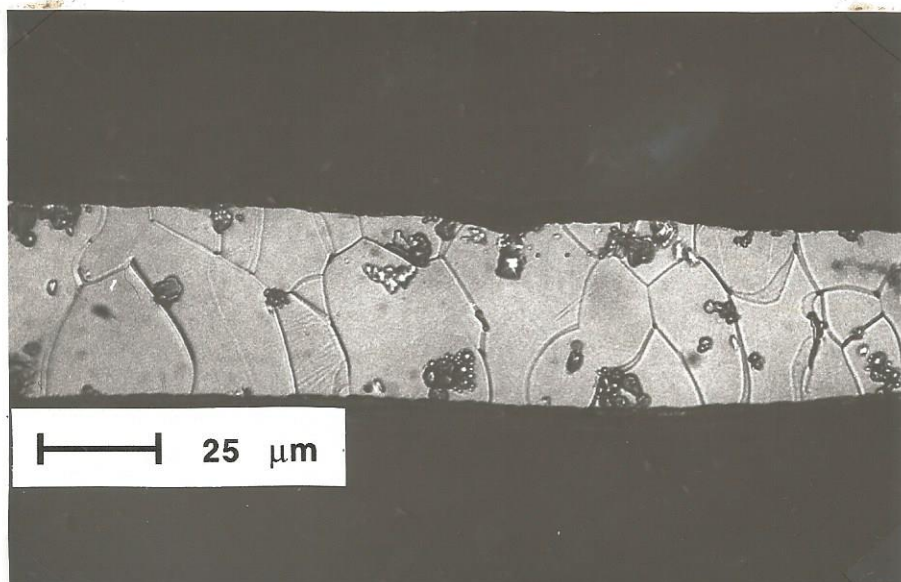
- * EQUIPMENT : As described in (3).[Figure E]
- * METHODOLOGY : Figure E
- * RESULTS OBTAINED : Figure F and Figures 1 (a, b) and 2 (a, b)

(3) B. Viala, J. Degauque, Thèse Doctoral, Laboratoire de Physique des Solides (C.N.R.S. – U.R.A. 74) I.N.S.A., Toulouse, 1994, 196 – 198.

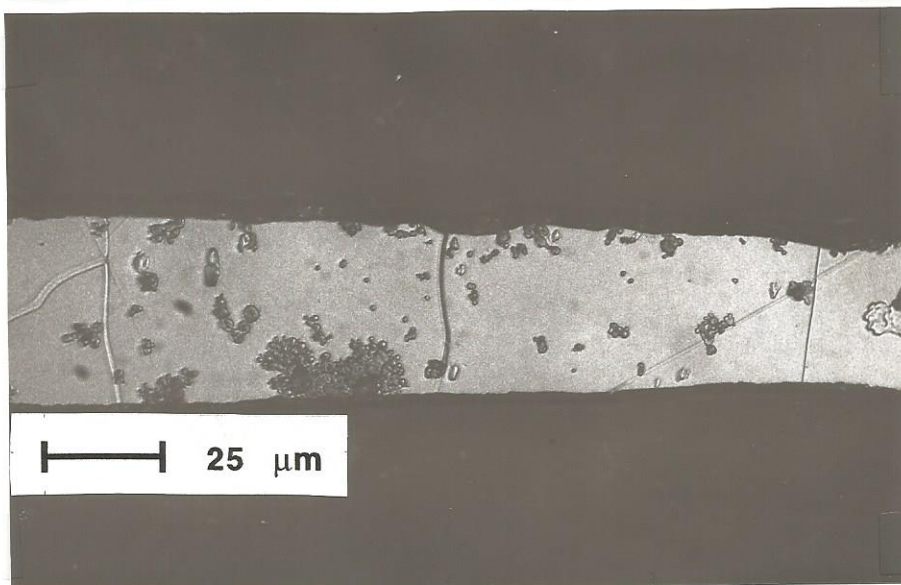
Table II: Grain Size and Texture

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

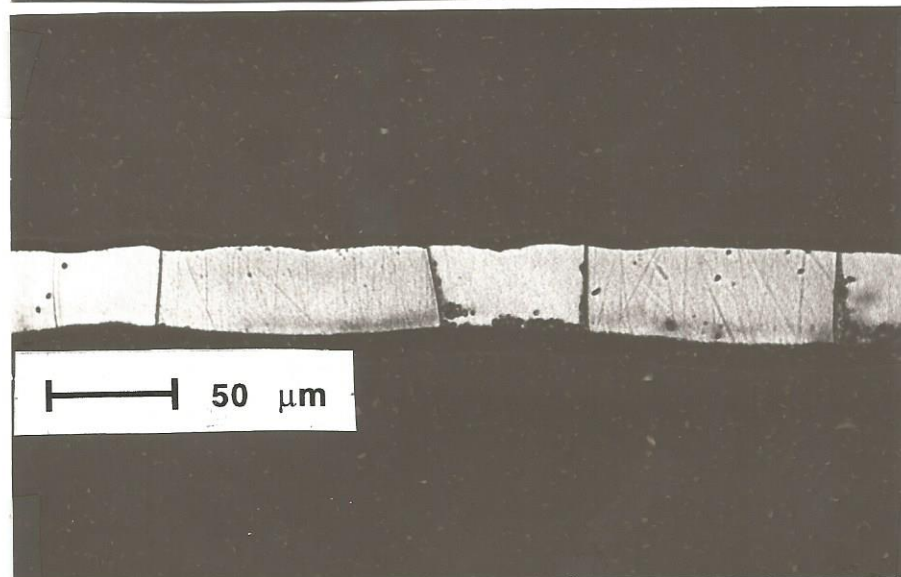
(D : Average grain size, t : thickness of the material , $1\mu\text{m} = 39.37\mu\text{in}$)



(a)
Cycle I
 $D < t$



(b)
Cycle I
 $D > t$



(c)
Cycle II
 $D > t$

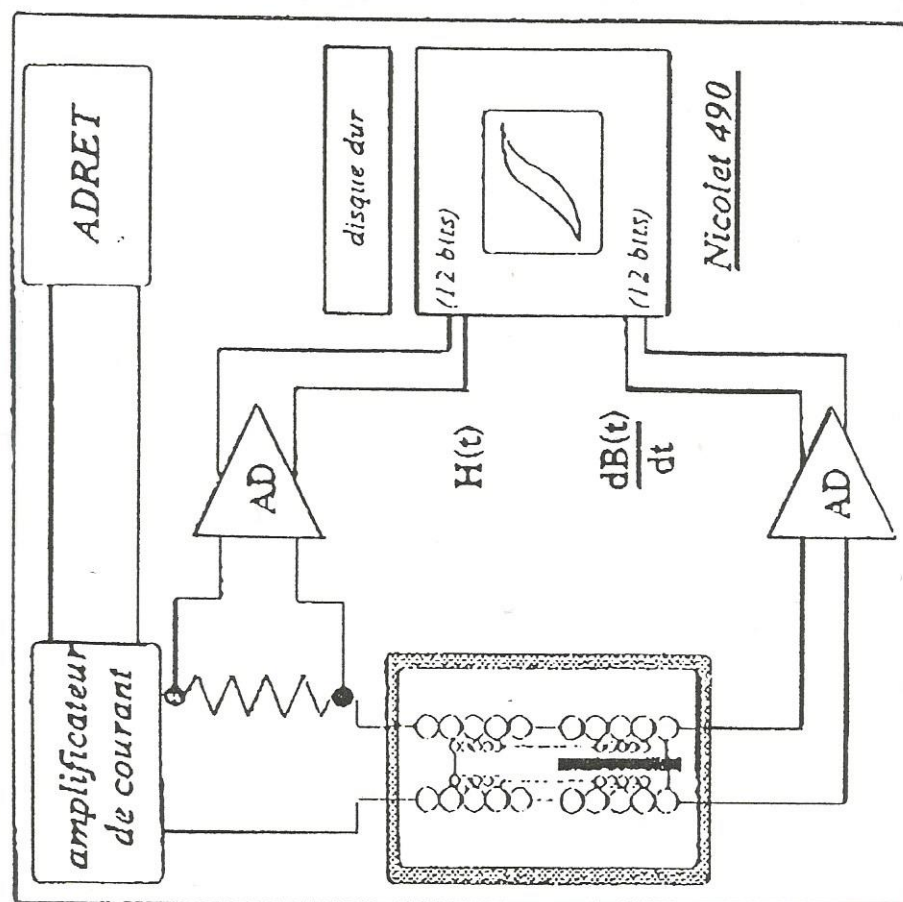
Figure D.- Micrographic structures (N_2)

a) Cycle I (900 °C), $D < t$, **b)** Cycle I, $D > t$, **c)** Cycle II (1100 °C), $D > t$

D : Average grain size, t : Material thickness

FIGURE E : EXPERIMENTAL EQUIPMENT (4)

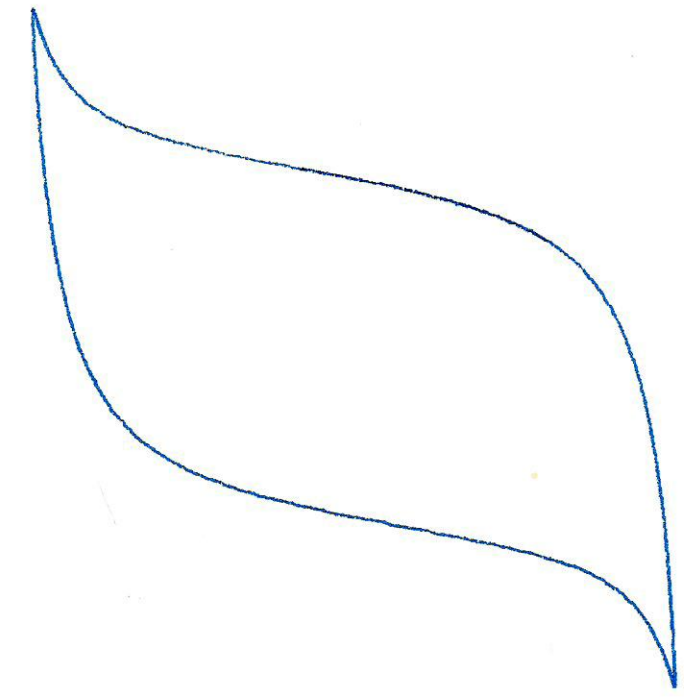
(4) B. Viala, J. Degauque, Thèse Doctoral, I.N.S.A. – C.N.R.S., Toulouse, (1994), pp 196 – 198.



Champ H

INDUCTION B

-- CYCLE D'HYSTERESIS --



CHAMP H

-100.0 A/m 0.0 A/m 100.0 A/m

ECHANTILLON :
N217
Bmax : 1.0151E+000 T.
Br : 8.6790E-001 T.
Hmax : 8.9520E+001 A/m.
Hc : 4.5040E+001 A/m.
PERTES: 1.0998E+001 W/kg.

f : 7.0000E+002 Hz.
Bobine Primaire.
K= 1.0000E+004 spires/m.
Bobine Secondaire.
n= 1.2000E+003 spires.
Resistance du montage.
R= 1.0000E+003 Ohms.

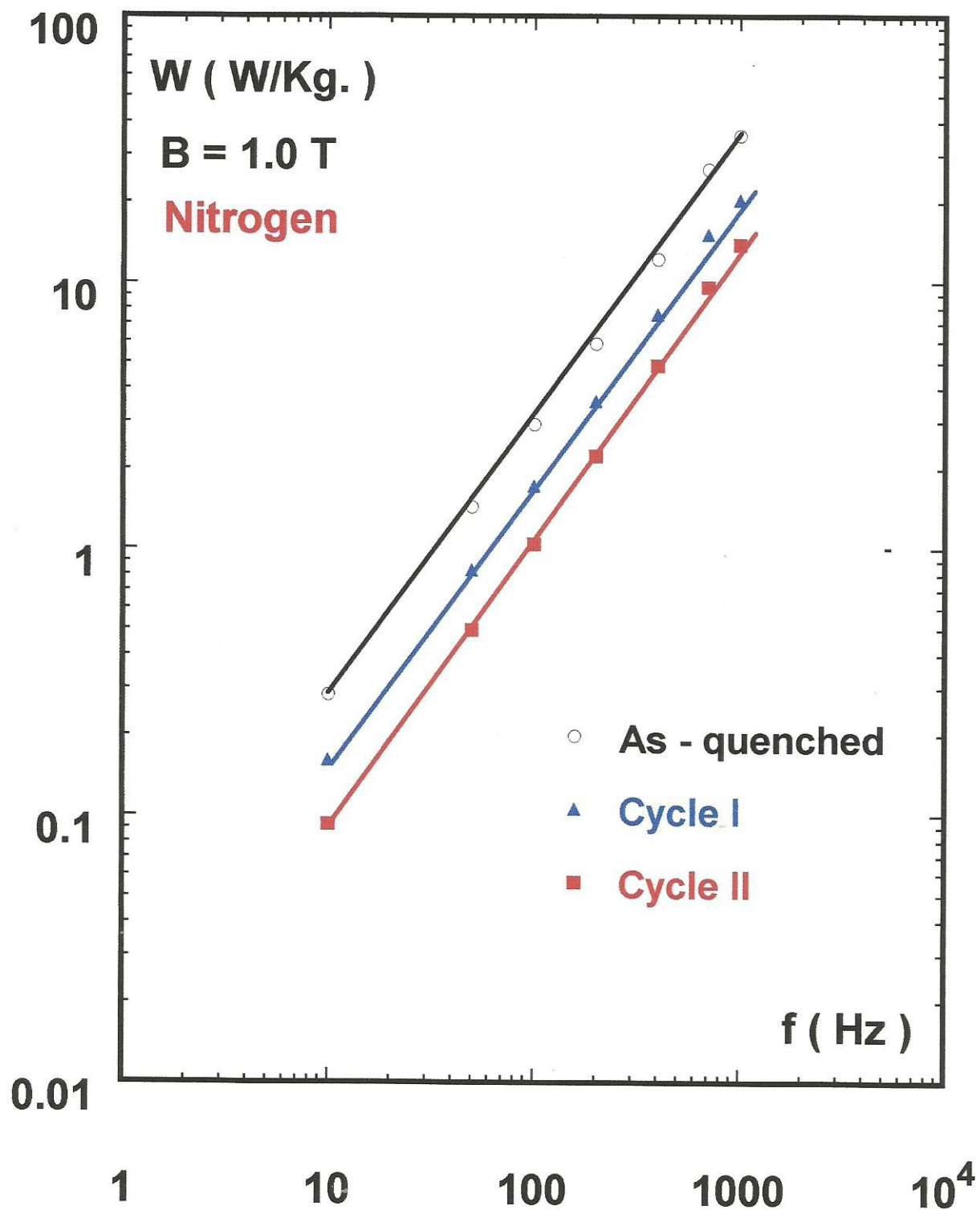


Figure 1 (a) : Influence of temperature on the amount of core losses. (1 W/Kg = 0.454 W/lb)

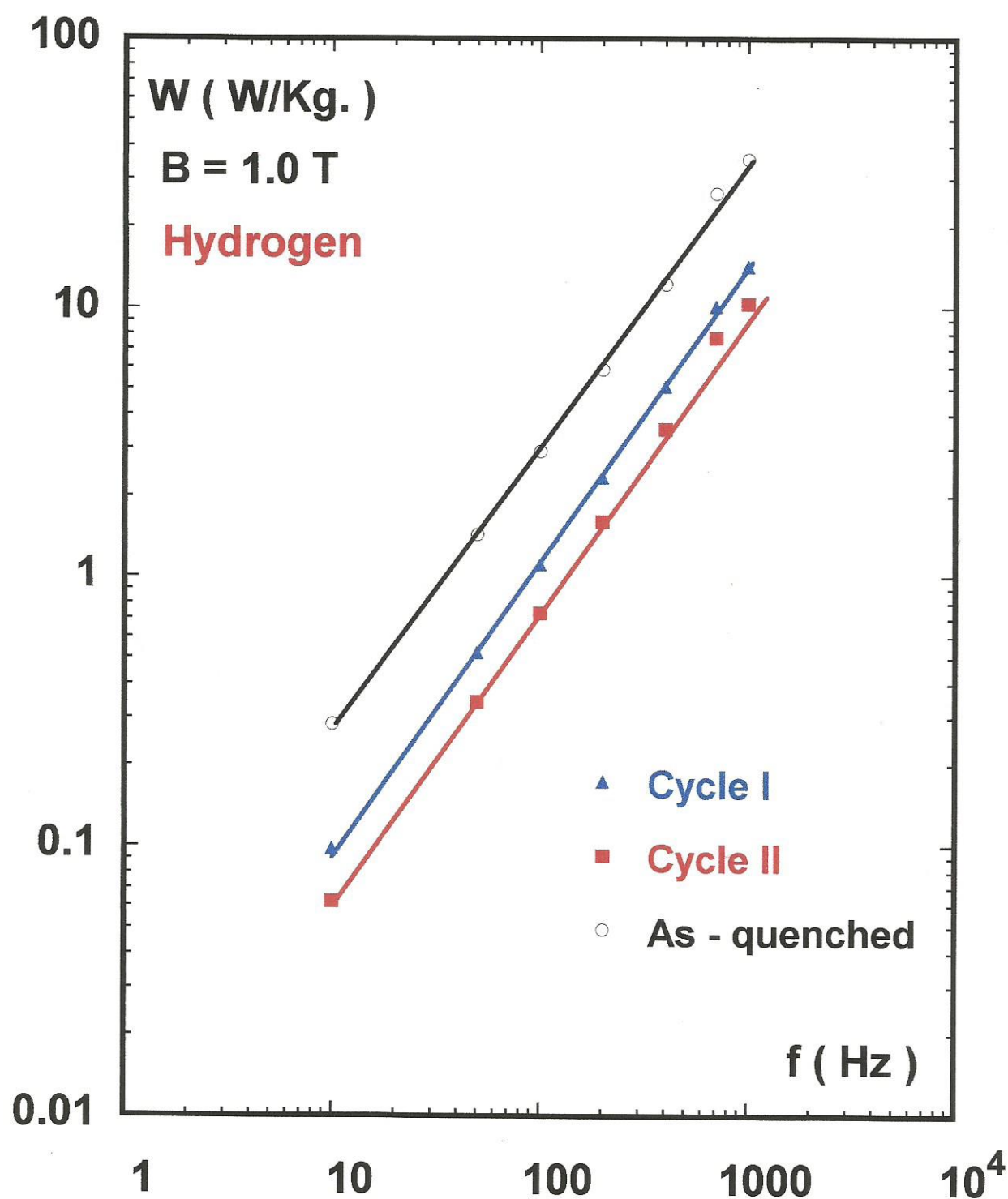


Figure 1 (b) : Influence of temperature on the amount of core losses. (1 W/Kg = 0.454 W/lb)

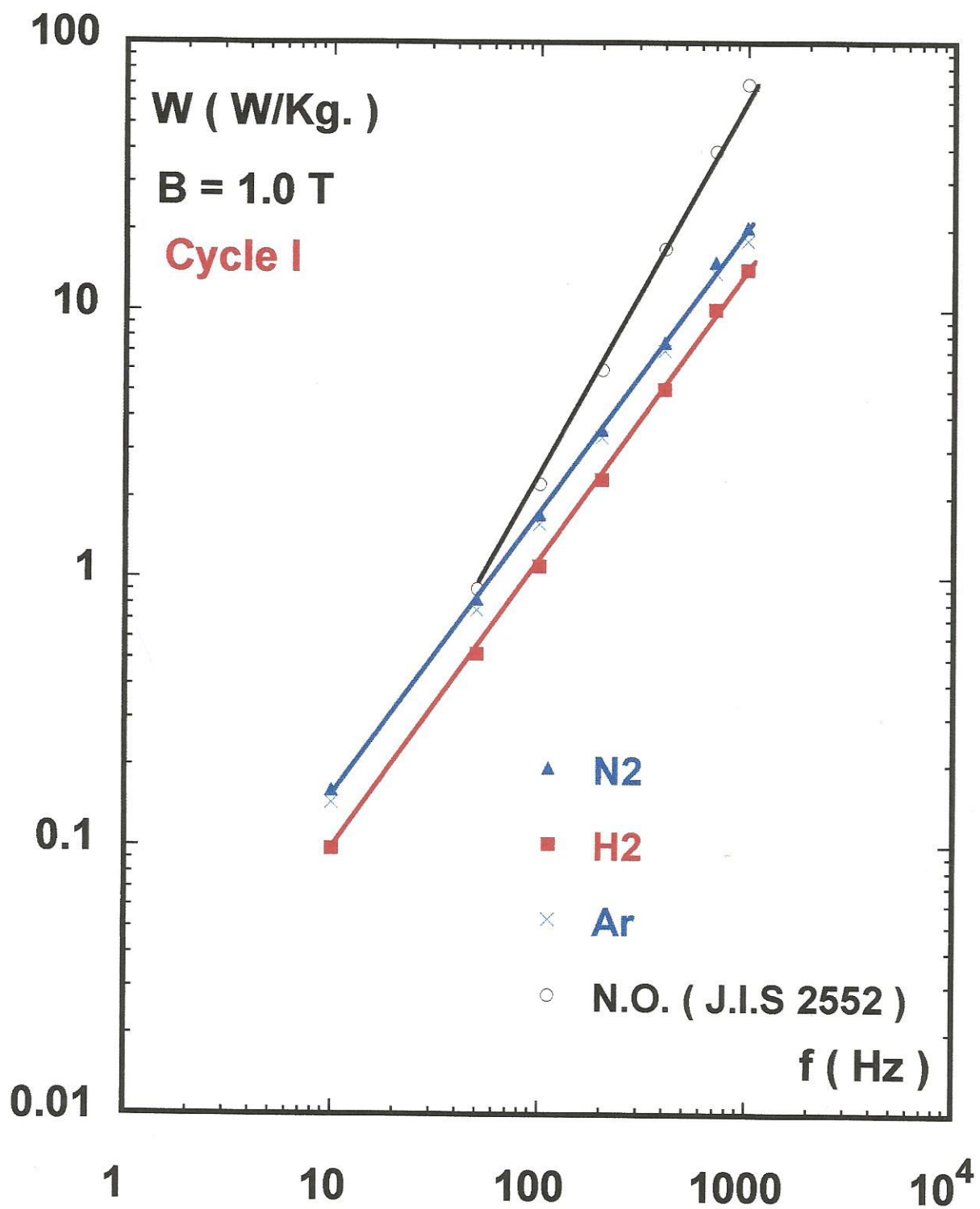


Figure 2 (a) : Influence of the atmosphere type on the core losses. (1W/Kg = 0.454 W/lb)

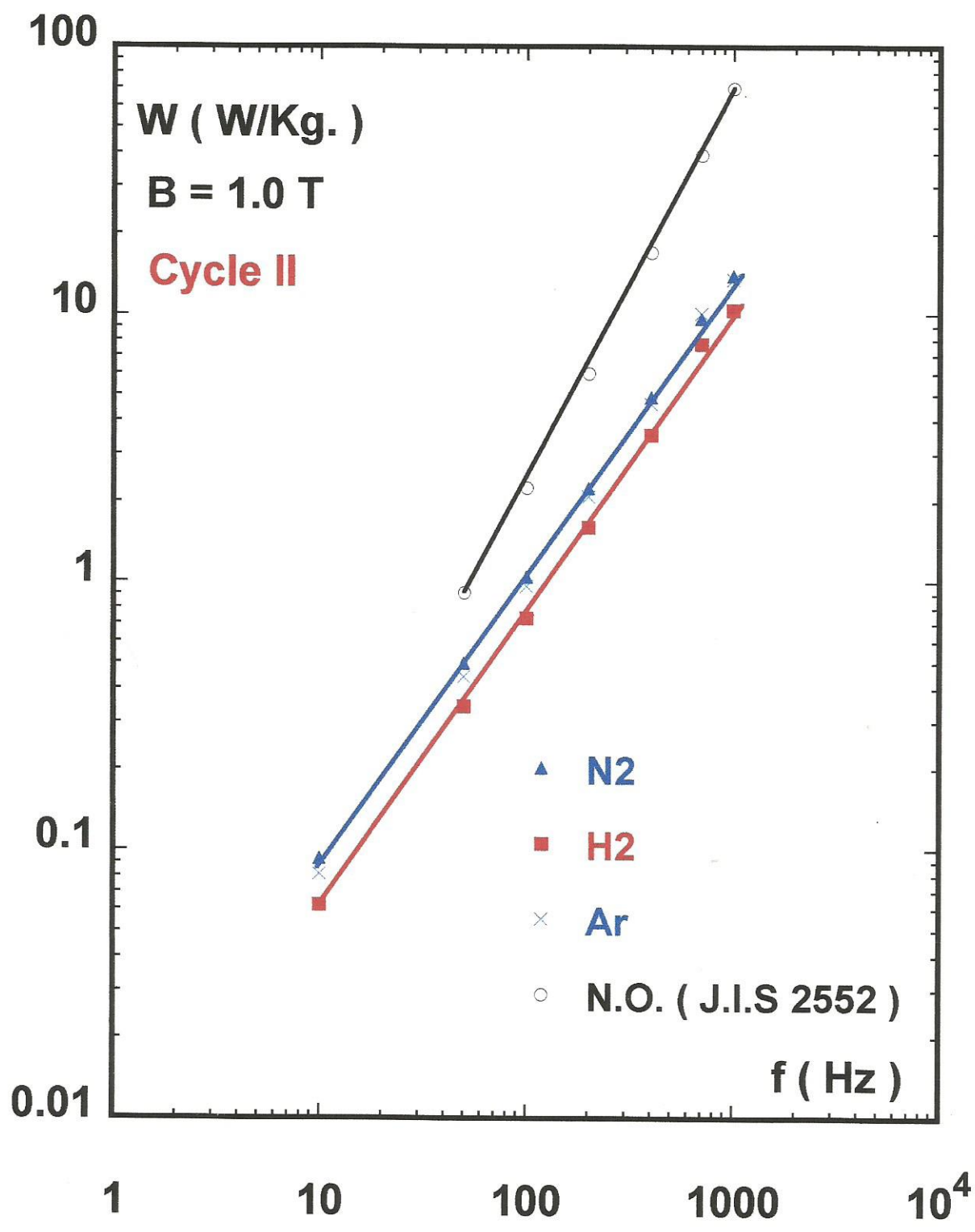


Figure 2 (b) : Influence of the atmosphere type on the core losses. (1W/Kg = 0.454 W/lb)

5.- RESULTS AND THEIR ANALYSIS.

➡ In view of the results obtained, the influence of the :

➔ Annealing temperature.

➔ Type of atmosphere.

➡ On :

➔ Grain size

➔ Texture

and



therefore

Magnetic
Characteristics
(core losses)

➡ Were analysed.

5.1.- INFLUENCE OF HEAT TREATMENT TEMPERATURE ON GRAIN SIZE, TEXTURE AND CORE LOSSES.

Grain size:

From the micrographic observations (Table II)
it can be deduced:

 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
larger

the material thickness (t).

 This fact suggests that an unfinished
recrystallization process has occurred under cycle I, in the
sense that residual stresses remain inside the material.

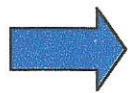
 Texture:

From the analysis of the data in (Table II) it can be deduced that :

 The component $\{ 310 \} < hkl >$ is predominant in all cases.

 That the complete elimination of the unwanted texture component $\{ 222 \}$, in relation to the as- quenched state, has been achieved.

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



Core Losses:



Figure 1 (a, b) shows the core losses at 1.0 T in the frequency range 10 – 1000 Hz. under nitrogen and hydrogen atmospheres.



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.

5.2.- INFLUENCE OF ATMOSPHERE TYPE ON GRAIN SIZE, TEXTURE 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.

➡ The difference in losses between N_2 and Ar atmospheres at (0.8 – 1.0 T) inductions varies between 5 – 10 % (Figure 2 (a, b)).

➡ In the case of hydrogen, there is an improvement in the { 200 } and { 110 } texture components, together with a notable increase in grain size.

➡ 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%).

6.- COMPARATIVE STUDY OF CORE LOSSES IN MAGNETIC MATERIALS OBTAINED BY DIFFERENT SIDERURGICAL PROCESSES.

➡ It must be pointed out that our results (core losses) are worse (Table III) than those obtained when the material is annealed under vacuum (1 – 20 Pa), where by means of :

A tertiary recrystallization process		D > 1000 μm (3,937 μin)
		Sharp texture {100}<0kl>

Can be obtained

➡ This type of annealing is more expensive than (N₂ or Ar) atmospheres.

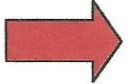
➡ Materials with high silicon content (6.5 %) obtained by conventional process + C.V.D. present magnetic characteristics similar to those obtained by means of Cycle I under N₂ or Ar atmospheres.

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Table III : Comparative Study of Core Losses in Magnetic Materials Obtained by Different Siderurgical Processes.

Material	Production Process	Heat Treatment	Thickness (μm)	Core Losses W/Kg.(1.0T)	
				f = 50 Hz.	f = 400 Hz
N.O.(6.5%Si)	Rap. Sol.	N ₂ 900 °C	38.2	0.83	7.58
“	“	N ₂ 1100 °C	“	0.49	4.90
“	“	Ar 900 °C	“	0.61	5.72
“	“	Ar 1100 °C	“	0.44	4.61
“	“	Vac.. 1050 °C	30 - 50	0.30 – 0.40	2.85 – 3.90
N.O.($\approx$ 3.2 %Si)	Conventional	Conventional	350 – 500	0.95 – 1.00	15.0 – 17.0
N.O.($\approx$ 3.2 %Si)	Conventional	C. V. D.	50 - 100	0.58 – 0.70	6.1 – 7.0

7.- CONCLUSIONS.

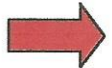
 The results obtained in the present paper allow us to state :

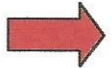
 The material obtained as it is described in this research work presents, in all cases, an amount of core losses inferior to that of the highest grade of conventional non-oriented magnetic steel sheet.

* Grade 35-A-250 [0.35mm (0.014 in) .J.I.S – 2552: 1986]

* Grade FeV-240-35HA (U.N.E.- E.N.- 10106 : 1996)

* Grade M-13 [0.35mm (0.014 in) A.I.S.I.]

 This becomes particularly important at frequencies above 200 Hz.

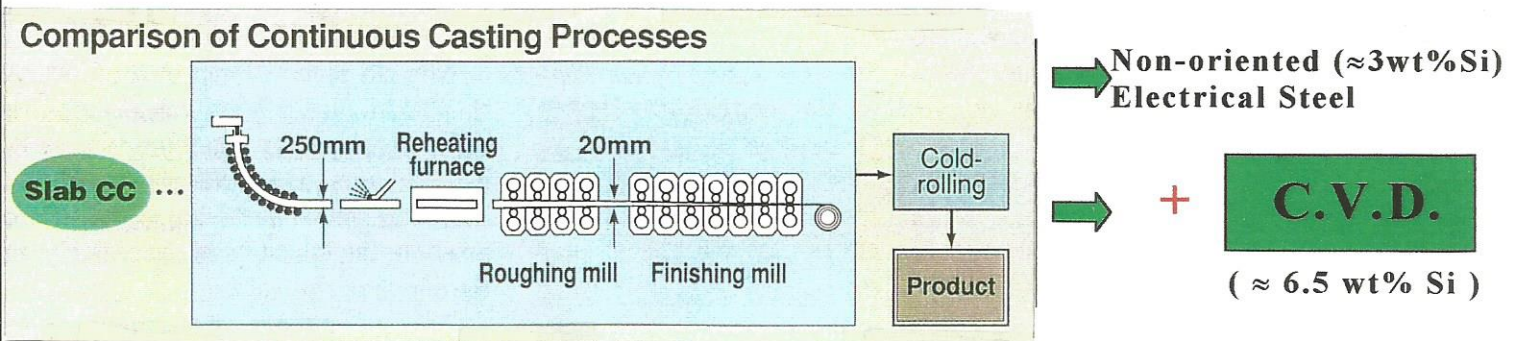
 In view of these results, for reasons of economy and ease of handling, the use of nitrogen atmospheres is recommended for the final heat treatment of these materials.

➡ The choice between annealing of type I [900 °C (1652 °F)] or II [1100 °C (2012 °F)] must be based on the comparison between :

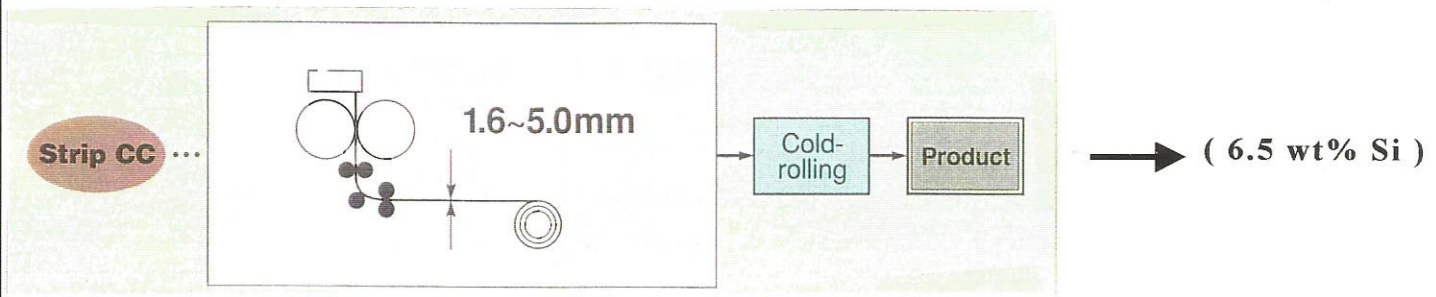
- * The improvement in the amount of total losses (30 – 40 %).
- * The higher cost and processing difficulties implied in the annealing temperature increase.

8.- NEW SIDERURGICAL PROCESS

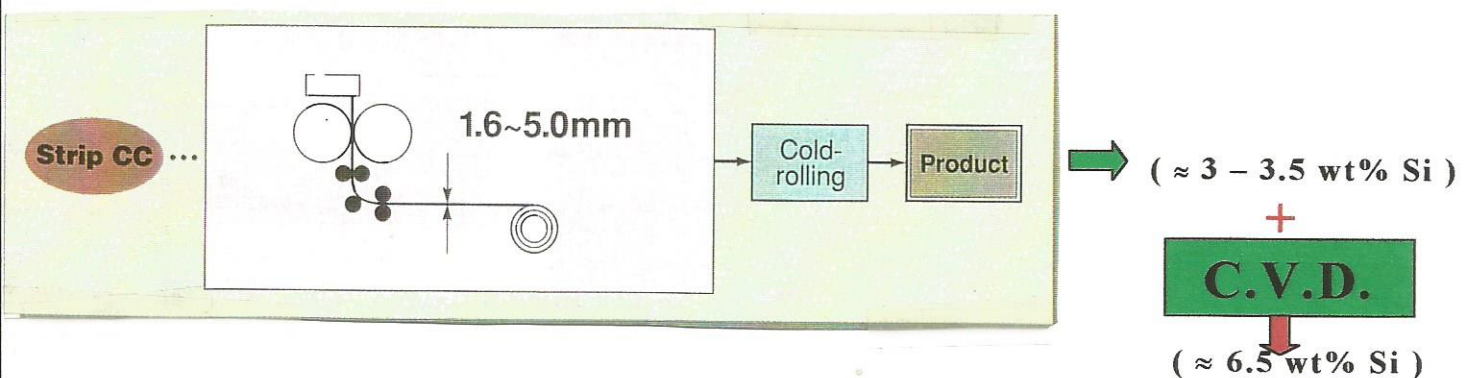
Conventional Process



The siderurgical process studied in this research.



New siderurgical process





Nippon Steel has completed the main cable erection for the Akashi Kaikyo Bridge (center span: 1,990 m, the world's longest). The company developed 180 kgf/mm² high-strength steel wire and used some 50,000 tons of it for the two main cables.

NIPPON STEEL NEWS

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NO. 256
MAY/JUNE 1996

World's First Twin-Drum Strip Caster to Be Installed at Hikari Works

Nippon Steel will install the world's first twin-drum strip caster at its Hikari Works to produce nickel stainless steel sheet.

It will be the world's first commercial application of the twin-drum strip caster which produces strip directly from molten steel, eliminating the need for a hot strip mill.

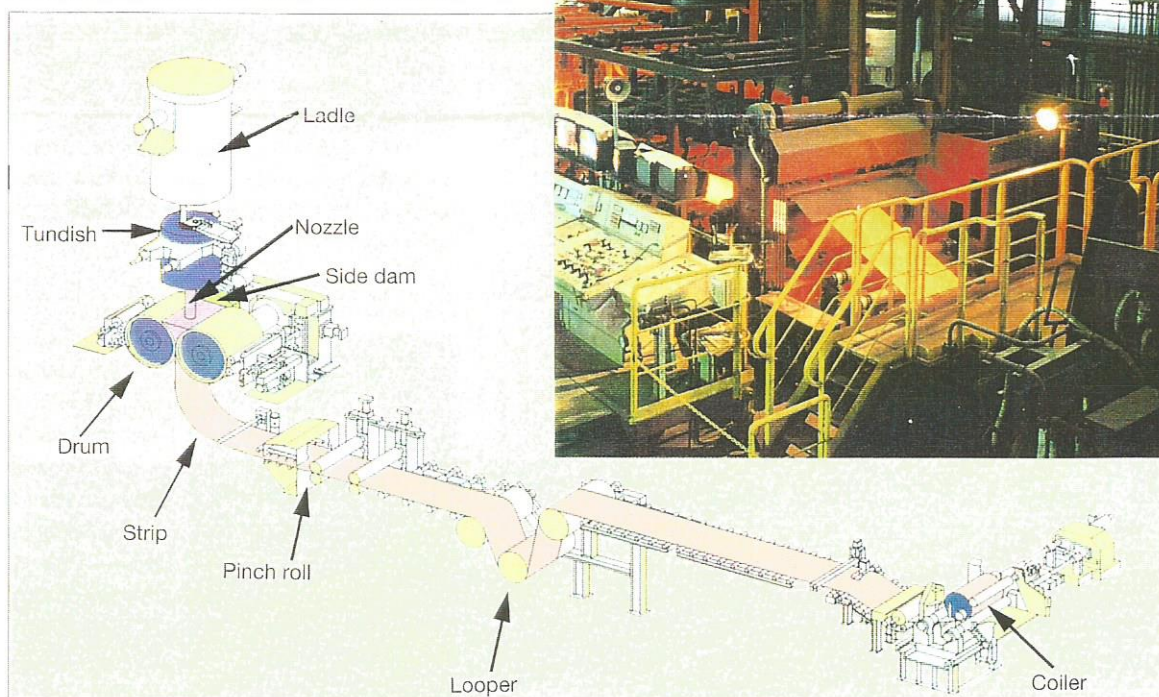
Construction of the strip caster, which will cost about ¥11 billion, started at the Hikari Works in January 1996 and is scheduled for completion in September 1997. It will have a production capacity of 35,000 tons a month, and strip sizes will be 2.0 to 5.0 mm in thickness and 760 to 1,330 mm in width.

In the twin-drum strip caster, molten steel from the tundish is poured into a pool enclosed by the two drums and two refractory side dams, solidified and extruded. As the strip emerges from under the drums, it is drawn by pinch rolls and coiled by a coiler. The inside surfaces of the drums are water-cooled and the outside surfaces are given a special coating to resist the high temperature of molten steel.

In 1986, Nippon Steel and Mitsubishi Heavy Industries, Ltd. started joint

research to develop the strip caster. A practical 10-ton twin-drum strip caster able to produce an 800-mm-wide strip was constructed, and test casting of nickel stainless steel was started in July 1989 (see *Nippon Steel News* No. 229).

Strip width was subsequently expanded to four feet, and technology for stable casting at this width was perfected. The quality of the final products (cold rolled sheets) was also confirmed to be equal to that of conventional products.



▲ Test casting with demonstration plant in 1991

◀ Twin-drum strip caster flow chart

9.- MAIN APPLICATIONS OF THIS MATERIAL AS A RESULT OF ITS MAGNETIC CHARACTERISTICS.

➡ Non – oriented material with a random texture,
low thermal losses and allowing down – size.

Applications



➡ Cores production for high performance electrical machines :

- * Electrical A.C.motors in aeronautics industry
(≈ 400 Hz.).

➡ Small (< 0.75 Kw) high speed induction motors
which work at frequencies between 50 – 200 Hz.

- * On-board equipments.

- * Hardware computers drivers.

Magnetostriction:

The magnetostriction is very small in any direction, which makes reduction of core noise possible (1).

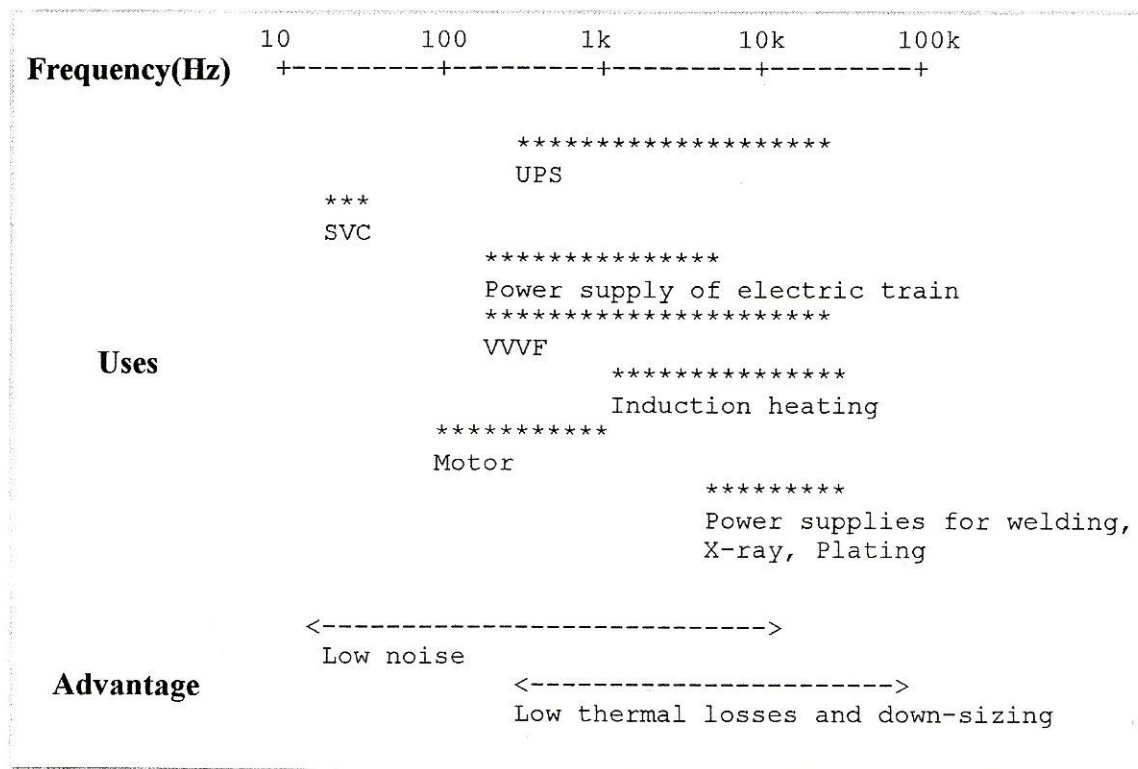
Applications



- ➔ Transformers operating at frequency range in electric power supply (200 – 10.000 Hz.) and uninterruptured power supply (U.P.S.).

(1) Y. Tanaka, et al., J.Magn. Magn. Mater., 83, (1990), 375 – 376.

Users and Advantage



10.- COMPLEMENTOS (I)

ORDER – DISORDER

(1) B. Viala, **J. Degauque**, M. Baricco, E. Ferrara, M. Pasquale, F. Fiorillo.

Magnetic and mechanical properties of rapidly solidified Fe – Si 6.5 wt % alloys and their interpretation.

J. Magn. Magn. Mater., 160, 1996, 315 – 317.

(2) D. Bouchara, M. Fagot, **J. Degauque**, J. Bras.

Ordering influence on magnetic properties of rapidly quenched Fe –6.5 wt% Si.

J. Magn. Magn. Mater., 83, 1990, 377 – 378.

(3) **J. Degauque**, M. Bouchara, M. Fagot, J. Brass, J. P. Redoules, Ph. Chomel, B. Astie.

Effect of order – disorder transition on magnetic properties of rapidly solidified high – silicon ribbons.

I.E.E.E., Trans. Magn., 26, 5, 1990, 2220 – 2222.

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(4) X. F. Bi, Y. Tanaka, K. Sato, K. I. Arai, K. Ishiyama, Y. Yamashiro.
The relationship of microstructure and magnetic properties in cold rolled
6.5% Si – Fe alloy.
I.E.E.E., Trans. Magn., 32, 5, 1996, 4818 – 4820.

(5) W. Ciurzynska, J. Zbrozczyk, J. Olszewski, J. Frackowiak, K. Narita.
Order – disorder effect on magnetic properties of rapidly quenched Fe –
6.5% Si alloy.
J. Magn. Magn. Mater., 133, 1994, 351 – 353.

(6) X. F. Bi, Y. Tanaka, K. Sato.
Effect of microstructure on the magnetic properties of 6.5% Si – Fe alloy.
J. Magn. Magn. Mater., 112, 1992, 189 – 191.

10.- COMPLEMENTOS (II)

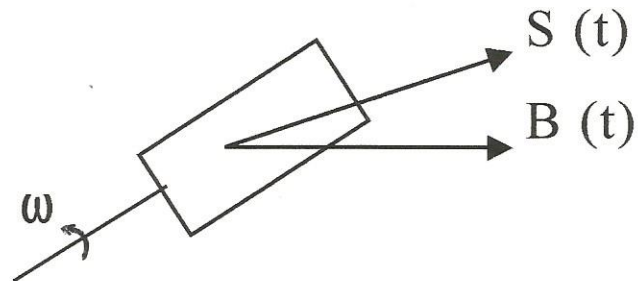
➔ Small (< 0.75 Kw) high speed induction motors which work at frequencies between 50 – 200 Hz.

$$V_s \text{ (synchronous speed)} = 60.f / p$$

f : frequency

p : number of field pole pairs.

10 – COMPLEMENTOS (III)



$$d\phi = \mathbf{B} \cdot d\mathbf{S} = B \cdot dS \cdot \cos \theta(t)$$

$$\phi = \int B \cdot \cos \theta(t) dS = B \cdot S \cdot \cos \theta(t)$$

$$\epsilon = - N \cdot (d\phi(t) / dt) = - N \cdot B \cdot S \cdot (d \cos \theta(t) / dt) =$$

$$= N \cdot B \cdot S \cdot \sin \theta(t) \cdot (d\theta(t) / dt) = N \cdot B \cdot S \cdot \omega \cdot \sin \theta(t) =$$

$$\epsilon_{\max} = N \cdot S \cdot B \cdot \omega = N \cdot S \cdot B \cdot 2 \cdot \pi \cdot f$$

$$\epsilon_{\text{rms}} = \epsilon_{\max} / \sqrt{2} = 4.44 \cdot N \cdot B \cdot S \downarrow f \uparrow$$

We can reduce $S \downarrow$ increasing $f \uparrow$

10.- COMPLEMENTOS (IV)

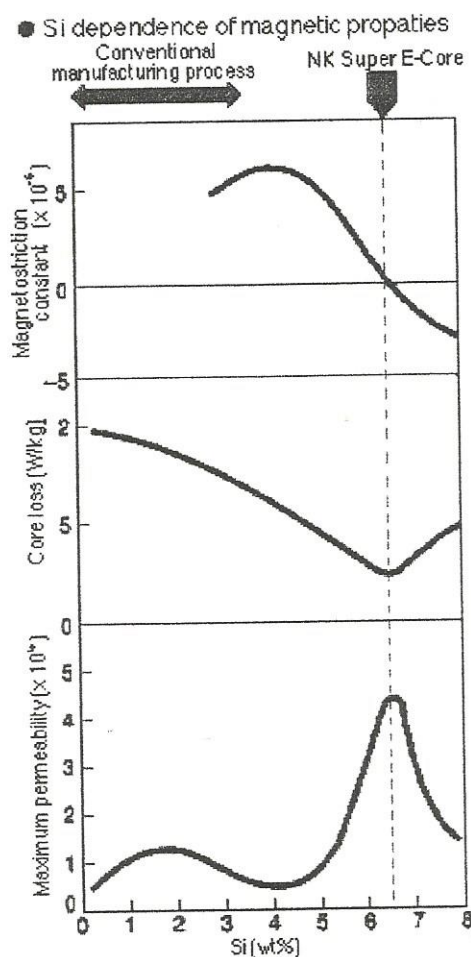


TABLE: Mechanical Characteristics

file:///A:/HTML/IGNAC~13.HTM

Mechanical Characteristics

Thicness(mm)	YP(N/mm ²)	TS(N/mm ²)	EI(%)	Vickers Hardness(Hv)
0.1	604	650	5.0	395
0.3	603	617	3.3	

e-core@lab.keihin.nkk.co.jp