

## **EFFECT OF COMPACTION TEMPERATURE ON MAGNETIC PROPERTIES OF IRON-RESIN COMPOSITES**

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### **ABSTRACT**

Soft magnetic materials produced by the P/M sintering technique are commonly used for DC applications. Iron-resin composites made by powder metallurgy show much better capability of machining and considerably lower eddy-current losses than laminated steels at medium and high frequencies. It is known that the magnetic induction and the permeability of iron-resin composites vary with the density. The warm compaction technology permits to increase the density thus increased maximum permeability and induction saturation may be expected by using that technique. Potential typical applications for this kind of materials are in automotive ignition cores, lighting ballasts, transformer cores, low frequency filters and chokes and electric motors.

In this paper, the mechanical and magnetic properties at low and high frequencies of iron-resin composite bars and rings made by cold and warm-pressing at different temperatures with and without curing are presented. The effects of these manufacturing conditions on the properties of the composites are discussed.

### **1. INTRODUCTION**

The powder metallurgy (P/M) technology is a well known process for the mass production of near net shape products in an economical manner. Many soft magnetic materials used for DC magnetic applications are produced by powder compaction and sintering of iron-based powder mixes (pure iron, iron-nickel, iron-phosphorus, etc.). New iron-based powder mixes are now available for AC magnetic applications. One of these products, ATOMET EM-1, is made of an iron powder admixed with a thermoset resin. The purity and high compressibility of the iron powder insures good ferromagnetic properties while the thermoset resin provides adequate mechanical properties and more importantly the necessary insulation between iron particles to minimize eddy currents. Soft magnetic composite materials can thus be produced using P/M compaction techniques followed by a low temperature curing treatment to cross-link the thermoset resin. This production technique reduces production costs, minimizes wasted products and allows to reach high density and good mechanical strength while maintaining precision of tolerance control compared to manufacturing of laminated steels. Iron-dielectric materials, in which the amount of resin is usually lower than 2 wt % [1], are also known as dielectromagnetics [2]. These materials are used in AC and pulsed DC electromagnetic systems [3]. Among the interesting properties of these soft magnetic composites, it is worth mentioning the isotropic magnetic and thermal properties providing a spatial distribution of magnetic flux and heat, the low AC magnetic core losses and their ease of manufacturing. They present many advantages over steel laminations that have anisotropic magnetic and thermal properties. Steel laminations are mainly restricted to low frequency applications (high core

losses at medium and high frequencies) and to simple geometries with magnetic flux in the plane of the sheet to avoid excessive eddy current losses [4].

Among potential applications of iron-resin composites, there are the replacements of costly lamination stack assemblies, brushless DC motors and micromotors, choke cores, linear tube asynchronous motors [5], cores of different electrical machines such as disc drive, rotary linear, ac homopolar, rotary cylindrical and linear tube induction motors [6]. The use of iron-resin composites to replace a costly lamination stack has recently been demonstrated [7]. There are also many other applications where the iron-resin composites will probably be used in the future. Designers, electrical and mechanical engineers should now rethink the concept of motors using laminations in order to most efficiently use the material and the fact that it is possible to have an isotropic distribution of the induction with the use of iron-resin composites. Design teams should take radical new design concepts. However to compete, iron-resin composites must show physical, mechanical and magnetic properties as good or better than those of stacked low carbon steel laminations. The warm pressing technology which uses heated powder in heated tooling during the compaction process, provides a means to produce P/M parts with high green densities in the 7.2-7.5 g/cm<sup>3</sup> range in a single step and to improve mechanical properties. Since the density has a great impact on magnetic properties (core loss, permeability, maximum induction, etc...), warm pressing is a promising technology to enhance these properties.

In this paper, some physical, electrical, mechanical and magnetic properties of iron-resin composites made by warm pressing, with or without a subsequent curing, are presented. Results and discussion of the effect of compaction temperature on DC and AC magnetic properties at low, medium and high frequency (up to 1 MHz) are also presented.

## 2. EXPERIMENTAL PROCEDURE

In order to study the effect of different compaction temperatures on physical, mechanical and magnetic properties of iron-resin composites, rectangular bars and rings were prepared by uniaxial compaction. The iron-resin mix composed of a high purity water-atomized iron powder (ATOMET 1001 HP) and 0.8 wt % of thermoset resin powder, was prepared by dry mixing in a V-cone blender for 30 minutes. The particle size of the iron powder is nominally less than 250  $\mu\text{m}$  while that of the resin powder is below to 150  $\mu\text{m}$ .

For each experimental condition, ten transverse rupture strength bars (3.175 x 1.270 x 0.635 cm) were compacted according to the MPIF Standard 41 and two ring specimens (5.08 o.d. x 4.45 i.d. x 1.27 cm) were also compacted in a floating compaction die. Bars and rings were pressed to 620 MPa (45 tsi) at room temperature (22°C), 65, 100, 120 and 140°C. There was no lubricant in the mixes and die wall lubrication with graphite spray was used for all compactations. The lubrication was first applied to die walls of the heated die and the powder was preheated in the die for a period of four minutes in order that the powder reached a uniform temperature before pressing. For each compaction temperature, half of the samples (bars and rings) were cured at 175°C for 1 hour in air. This curing was done to fully cross-link and bind the resin to particle surfaces in order to increase the mechanical strength of specimens.

Density (measured from weight and physical dimensions of parts), electrical resistivity and transverse rupture strength (TRS) (MPIF standard 15) were evaluated before and after curing. Electrical resistivity was measured using a commercial micro-ohmmeter equipped with a four-point probe (0.8 cm between contact points). Ten resistivity measurements were taken on each rectangular bar before and after curing and averaged: five on one side and five on the other side in the compaction direction.

Magnetic measurements for DC and AC characterization at low and medium frequencies were made using a SMT/ACT-500 computer-automated magnetic hysteresisgraph. For DC magnetic characterization, rings were wound with 600 primary turns of #24 gauge insulated copper wire and 150 secondary turns of #30 gauge copper wire. For AC characterization at low and medium frequencies, each ring was wound with 250 primary turns of #24 gauge insulated copper wire and 250 secondary turns of #30 gauge copper wire. At high frequency, the apparent permeability was evaluated with a HP 4192A LF impedance analyzer and with a HP 16047A test fixture. The high frequency characteristics were evaluated from 1 kHz to 1 MHz. The excitation was 1 V<sub>rms</sub> producing an induction field  $B < 5 \times 10^{-4}$  T. For the characterization at high frequency, rings were wound with 96 turns of #24 gauge insulated copper wire. The specific losses or core losses  $P(\omega)$  were evaluated at 0.001 T (10 G) using the following relation:

$$p(\omega) = \oint \text{HdB} = \frac{1}{2\mu_0} \omega \mu'' \left| \frac{B^*(\omega)}{\mu^*(\omega)} \right|^2 \quad ,(\mu\text{W}/\text{cm}^3) \quad (1)$$

$$p(\omega) = \frac{0.025 \cdot f(\text{Hz}) \cdot \mu'' \cdot B^2 (\text{G})}{\mu'^2 + \mu''^2} \quad ,(\mu\text{W}/\text{cm}^3)$$

where  $\omega (=2\pi f)$  is the frequency,  $\mu_0$  ( $4\pi \times 10^{-7}$  H/m) is the permeability of free space,  $\mu'$  and  $\mu''$  are respectively the real and imaginary part of the complex permeability  $\mu^*(\omega)$  and  $B^*(\omega)$  the induction. This relation is valid only at low harmonic applied field which is the case for 0.001 T (10 G) induced field and mathematically represents the integral of the applied field H from the extreme tips of the hysteresis loop.

### 3. RESULTS AND DISCUSSION

#### 3.1 Physical, mechanical and electrical properties

The effect of the compaction temperature on density of iron-resin rectangular is presented in Figure 1. The densities, before and after curing, increase with the compaction temperature for all samples. This can be attributed to an increase of the ductility and a reduction of strain hardening of iron particles during compaction in the heated die [8]. For instance, a density of 7.35 g/cm<sup>3</sup> is reached on bars warm-pressed at 120 and 140°C. This corresponds to 97.7% of the theoretical density of the specimen (7.53 g/cm<sup>3</sup>). However, the curing does not significantly affect the density of samples in the range of compaction temperatures studied. In preliminary tests [9], it had been observed that the density slightly decreases above 140°C. This was likely caused by cross-linking in the resin during the powder heating before compaction. Figure 1 also shows that the density of bars is systematically higher than that of rings. This is probably caused by a higher friction in the die during compaction of rings that have larger sliding surfaces than rectangular bars. The pressure transfer from punches is less efficient for a ring than for a rectangular bar because the surface in contact with die walls is approximately three times larger for a ring. It should also be noted that the evaluation of density is more accurate for rectangular bars (5 samples for each experimental condition) than for rings (2 samples for each experimental condition).

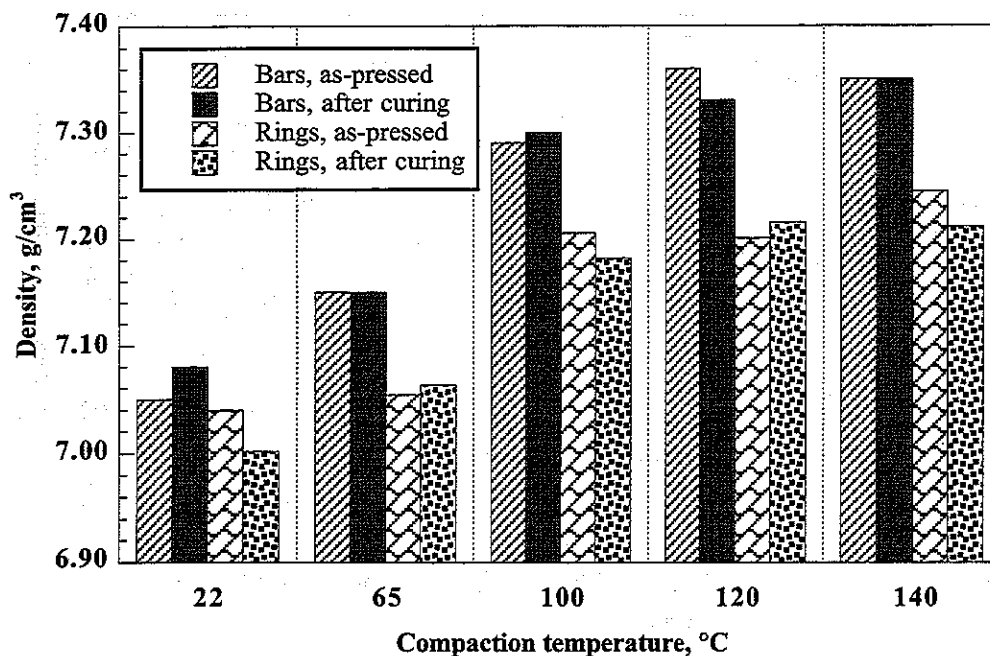


Figure 1: Effect of compaction temperature on density of rectangular bars and rings pressed at 620 MPa (45 tsi). (As-pressed or after curing at 175°C for one hour)

The effect of the compaction temperature on TRS is presented in Figure 2. For as-pressed specimens, the strength increases with the compaction temperature, while for cured specimens strength is relatively high for low compaction temperatures (116 to 130 MPa) and decreases for compaction temperature higher than 120°C (90 to 95 MPa). The TRS of as-pressed specimens principally increases because the density increases with compacting temperature as shown in Figure 3. Below a compaction temperature of 100°C, the curing treatment provides a large increase in strength while above that temperature the gain is less important. Consequently, for certain applications where a high density is needed, warm-pressing at a temperature above 100°C can provide both high density and high strength without curing.

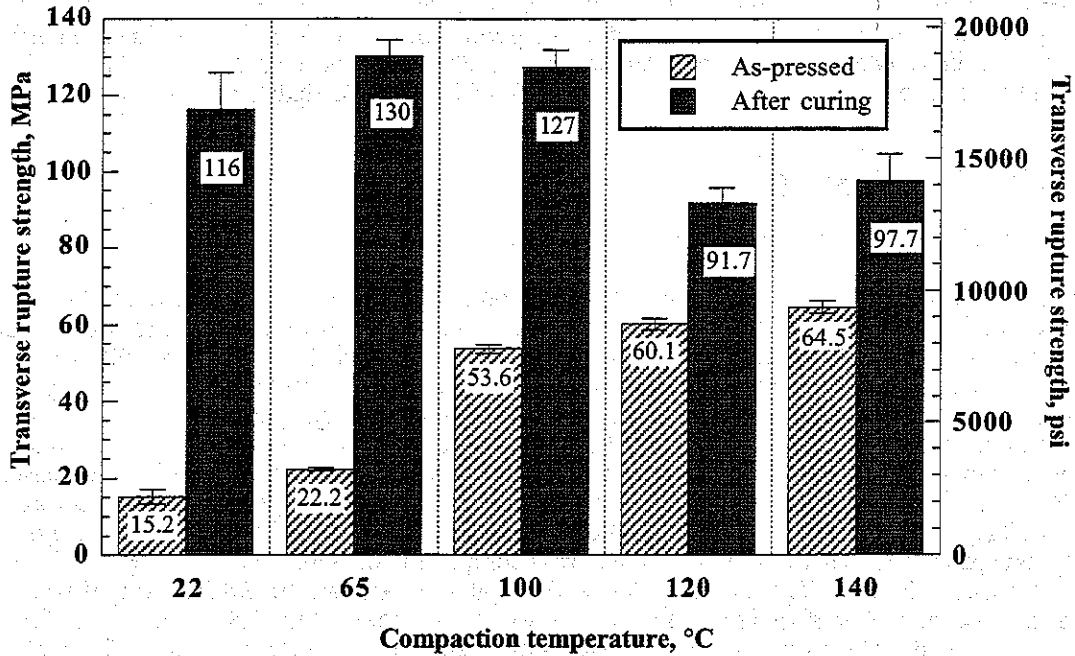


Figure 2: Effect of compaction temperature on transverse rupture strength of rectangular bars pressed at 620 MPa (45 tsi). (As-pressed or after curing at 175°C for one hour)

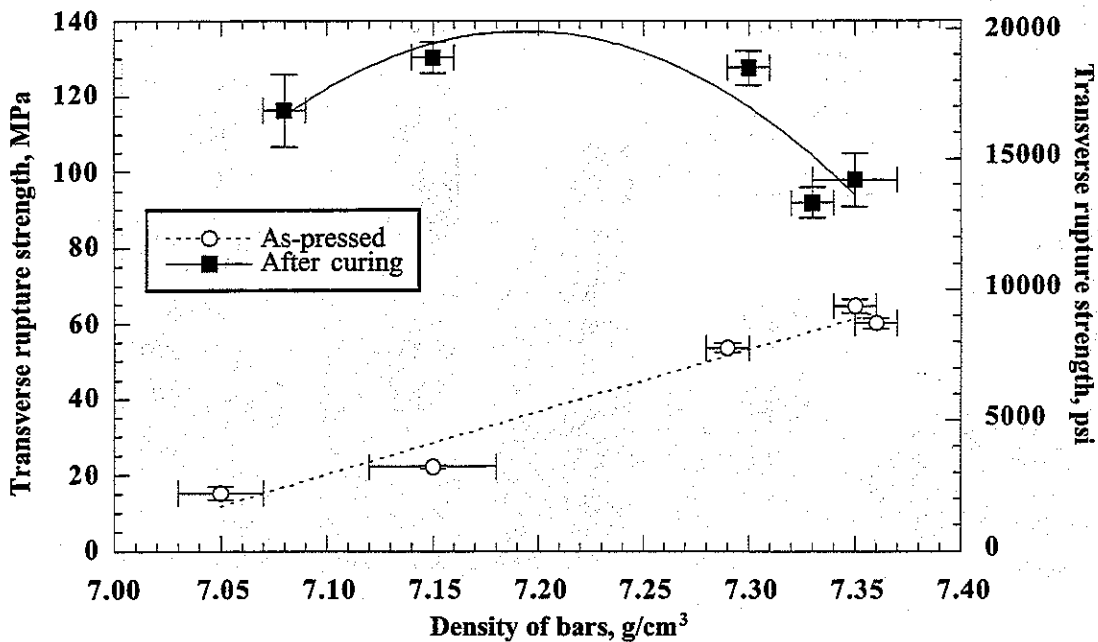
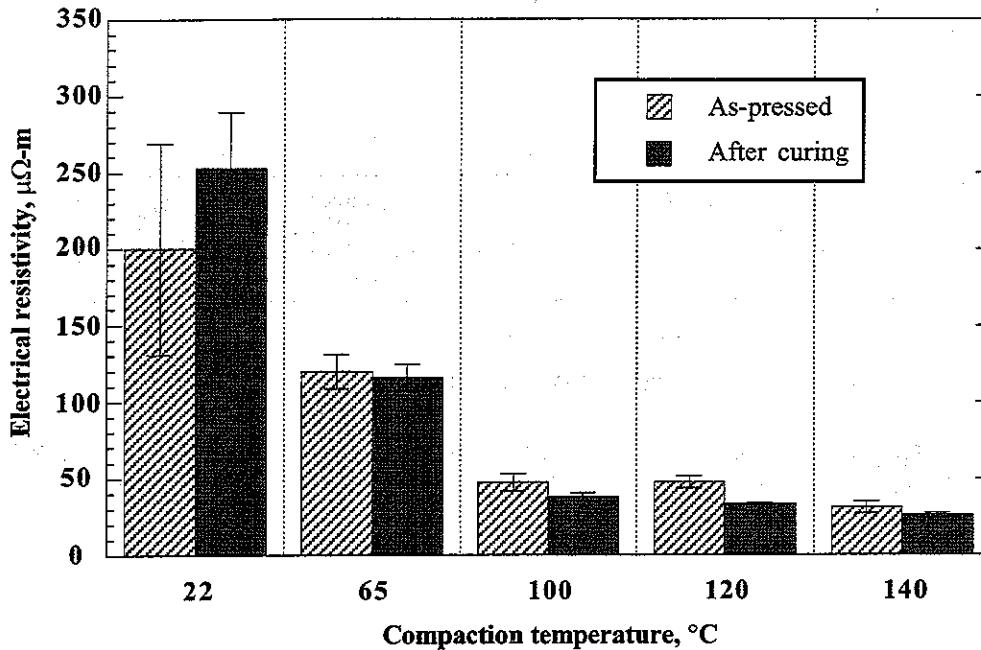


Figure 3: Transverse rupture strength as a function of the density of bars pressed at 620 MPa (45 tsi) and at different temperatures. (As-pressed or after curing at 175°C for one hour)

The effect of compaction temperature on the electrical resistivity of bars is presented in Figure 4. As expected, the electrical resistivity rapidly decreases with the compacting temperature because the number of metal to metal contacts between iron particles increases. This figure also shows that there is no significant difference at 95% confidence interval between the electrical resistivity measured before and after curing. The larger standard deviations for specimens pressed at 22°C suggest that the distribution of the resin is less homogeneous or uniform in these specimens than in those compacted at higher temperature. It is believed that the resin flows better during compaction at temperatures higher than 22°C.



**Figure 4: Effect of compaction temperature on electrical resistivity of rectangular bars pressed at 620 MPa (45 tsi). (As-pressed or after curing at 175°C for one hour)**

### 3.2 DC magnetic properties

Plots of the maximum induction  $B_{max}$ , maximum permeability  $\mu_{max}$ , residual induction  $B_r$  and coercive field  $H_c$  as a function of the compaction temperature for different applied fields  $H$  are presented in Figures 5 to 8 respectively. Generally, characteristics that make soft magnetic materials good are a narrow hysteresis loop (low  $H_c$  and high  $\mu_{max}$ ) and high  $B_{max}$ . Low residual induction,  $B_r$  is also suitable in order to avoid induced reverse current during the application cycles [10]. Since these characteristics are generally improved with an increasing density, warm-pressing could be an interesting alternative to regular compaction at room temperature. As shown in Figure 5, maximum induction increases with increasing compaction temperature. A maximum induction close to 1 T is reached at an applied field of 3980 A/m (50 Oe). At 9950 A/m (125 Oe) and 19900 A/m (250 Oe) applied fields, maximum inductions of 1.35 and 1.55 T are reached respectively for compaction temperatures in the 100 to 140°C range. Maximum permeabilities also increase by increasing the compaction temperature and values in excess of 300 can be reached by warm-pressing (see Figure 6). However, residual inductions and coercive forces are virtually not affected by varying the compaction temperature and values near 0.24 T and 420 A/m respectively are obtained. (Figures 7 and 8).

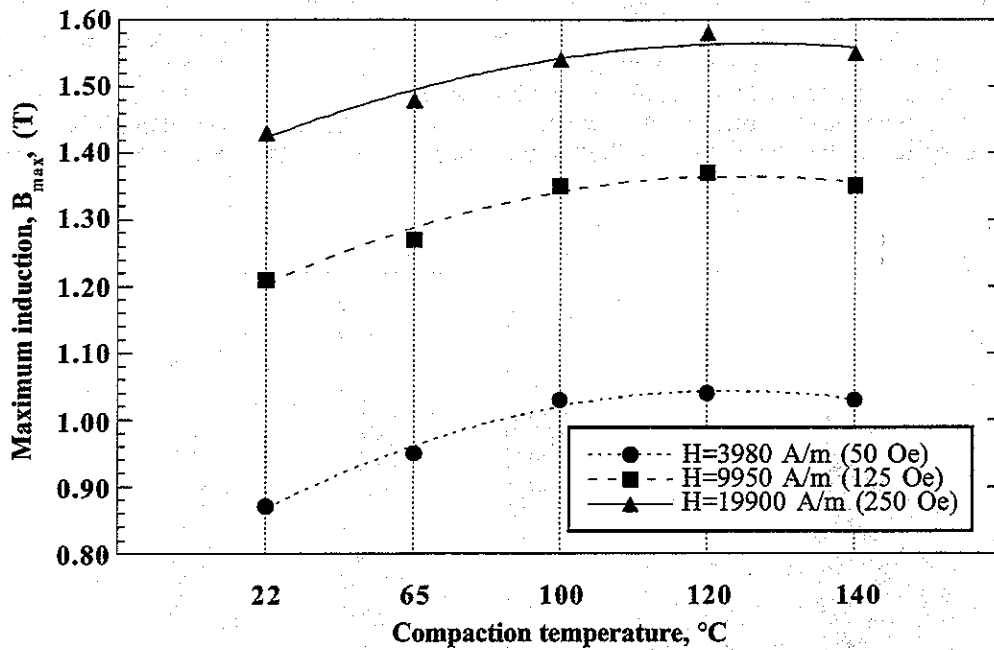


Figure 5: Effect of compaction temperature on maximum induction at different applied fields of rings pressed at 620 MPa (45 tsi). (After curing at 175°C for one hour)

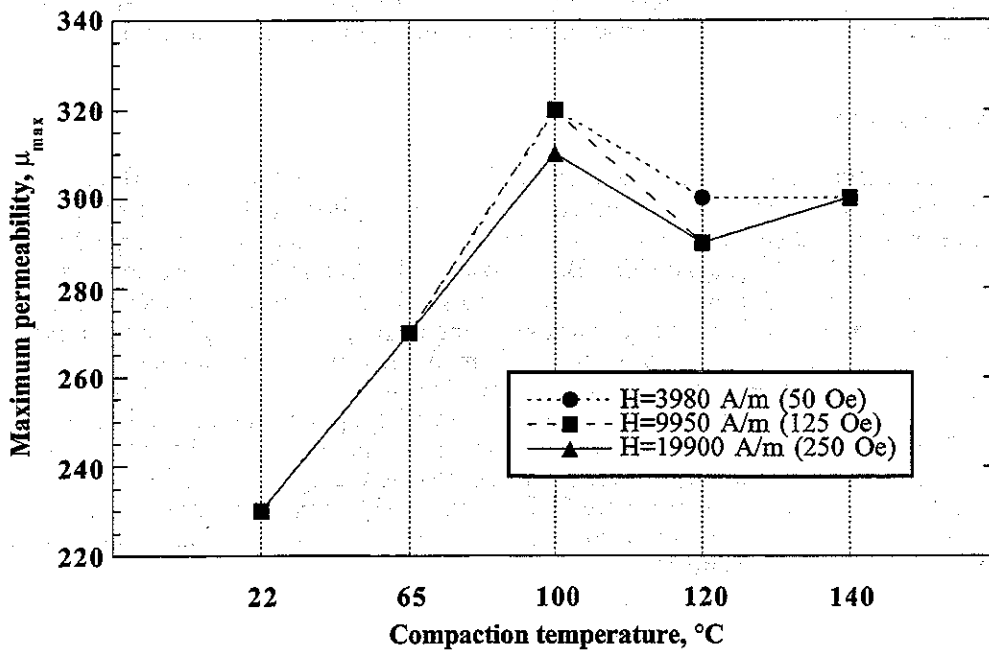


Figure 6: Effect of compaction temperature on maximum permeability at different applied fields of rings pressed at 620 MPa (45 tsi). (After curing at 175°C for one hour)

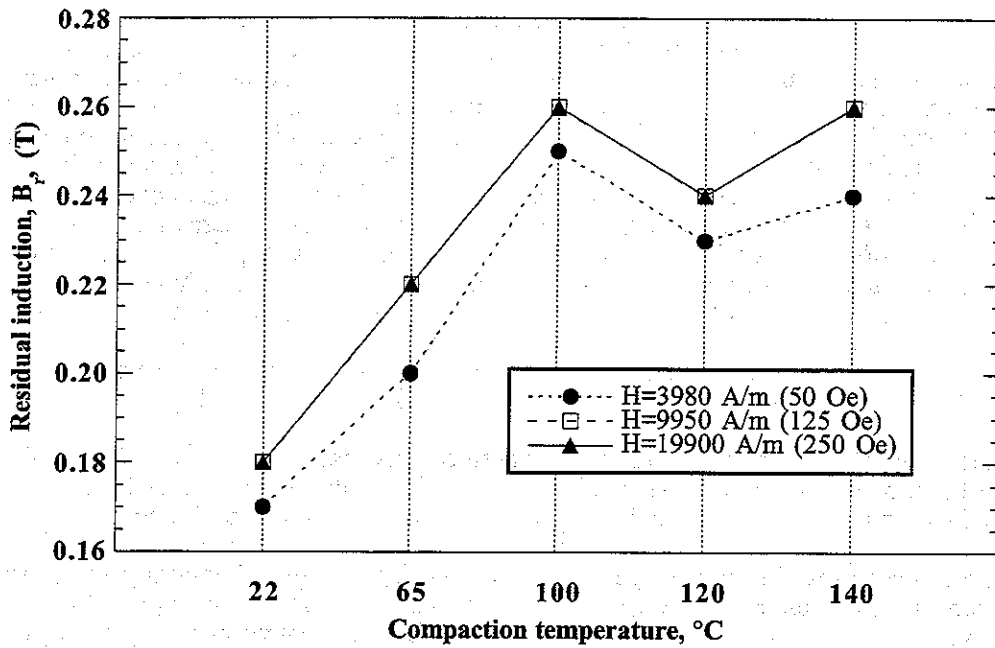


Figure 7: Effect of compaction temperature on residual induction at different applied fields of rings pressed at 620 MPa (45 tsi). (After curing at 175 $^{\circ}\text{C}$  for one hour)

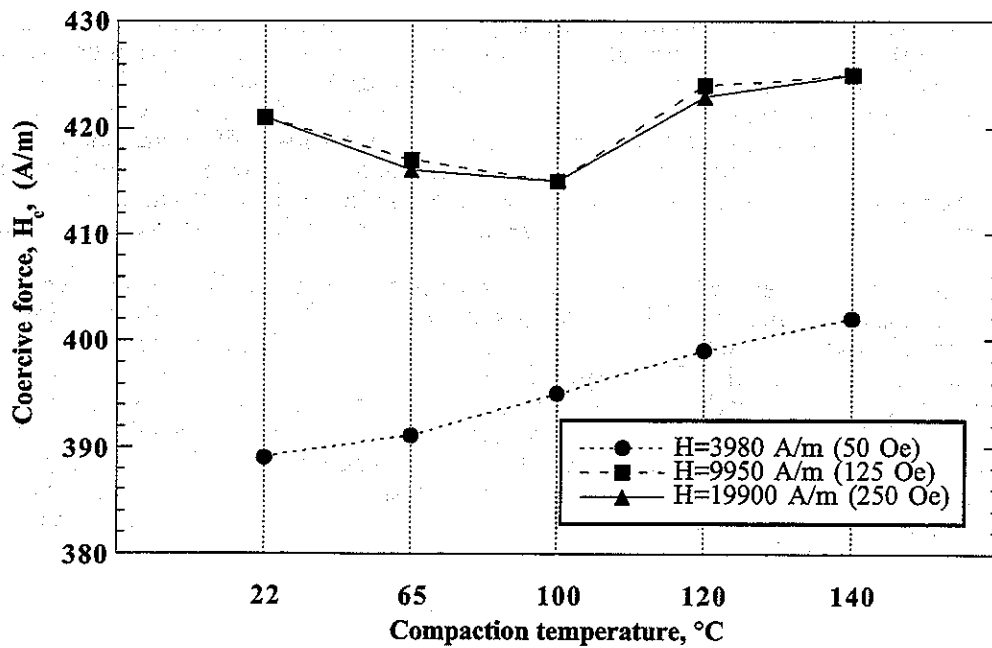


Figure 8: Effect of compaction temperature on coercive force at different applied fields of rings pressed at 620 MPa (45 tsi). (After curing at 175 $^{\circ}\text{C}$  for one hour)

### 3.3 AC magnetic properties at low and medium frequencies

The effect of compaction temperature and curing treatment on apparent core losses of iron-resin composite materials is shown in Figure 9. There is no significant difference in core losses measured on as-pressed or cured rings at both 60 Hz (Fig. 9a) and 400 Hz (Fig. 9b). It has been noted in Figures 1 and 4 that density increases and resistivity decreases with an increase of the compaction temperature. Since an increase in the density decreases hysteresis loss and a decrease in the resistivity results in an increase of the eddy current loss, experimental results show that they probably cancel each other when the compaction temperature increases. Nevertheless, as expected, it is shown that apparent core losses increase with both frequency and magnetization. In fact, apparent core losses increase because eddy currents increase with the square of frequency and the magnetic induction [11]. The average core losses at 60 and 400 Hz are respectively 11 and 79 W/kg for a magnetization of 1.0 T. These values are as good or better than those obtained with carbon steel laminations (1008 or 1018). For instance, the average core loss of a 1008 carbon steel laminations at 60 and 400 Hz are respectively 10 and 180 W/kg at 1.0 T magnetization [12]. At a magnetization of 1.5 T and 400 Hz, the iron-resin composite performs better than a 1018 steel lamination with core losses of 130 and 200 W/kg respectively [13].

The effect of the compaction temperature on apparent permeability measured at 60 and 400 Hz for as-pressed or cured iron-resin composites is shown in Figure 10. Permeabilities at 60 and 400 Hz for as-pressed or cured specimens are approximately the same, thus not affected by the frequency. For as-pressed specimens, the apparent permeability increases with the compaction temperature, reaches a maximum and then decreases. This is mostly related to the density of rings. After curing, the apparent permeability increases with compaction temperature. It seems that a certain stress relief of the iron occurs during curing even at this low temperature (175°C or 350°F). The apparent permeability of rings pressed at 120 and 140°C increases by approximately 10 and 25% respectively after curing. The apparent permeability decreases with an increase of the magnetization. In fact, for this range of frequencies, as the applied field increases, eddy currents increase and induce an opposing magnetization which decreases the overall effective magnetization thus also decreasing the permeability. The maximum apparent permeabilities are achieved for high compaction temperatures (100-140°C) after curing: values of 500 and 400 are reached for inductions of 0.5 and 1.0 Tesla respectively.

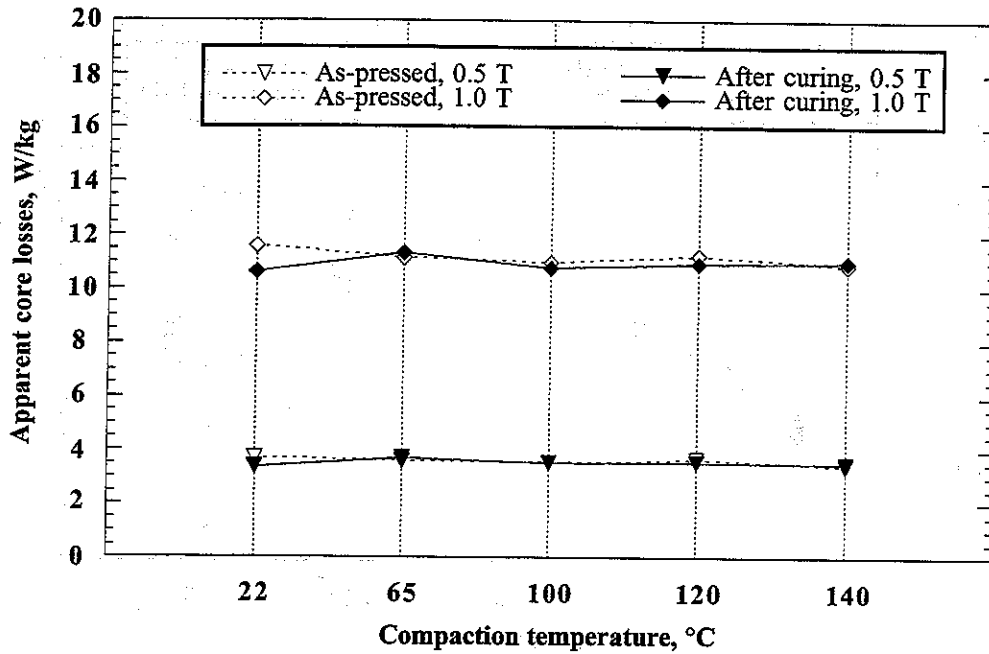
### 3.4 AC magnetic properties from 1 kHz to 1 MHz

The effect of the frequency on initial permeability of rings pressed at different temperatures is illustrated in Figure 11. Initial permeabilities, measured at very low induction (0.001 T), are constant up to a certain frequency range over which they start to drop with further increase of the frequency. Figure 11 also shows that the initial permeability increases with the compaction temperature: 75 for rings pressed at room temperature, 80 at 65°C, and approximately 85 for rings pressed in the 100-140°C compaction temperature range. This increase in initial permeability is attributed to the increased density. On the other hand, the better the insulation between iron particles, the higher the frequency at which initial permeability drops. This is mainly caused by eddy currents that increase with the square of the frequency.

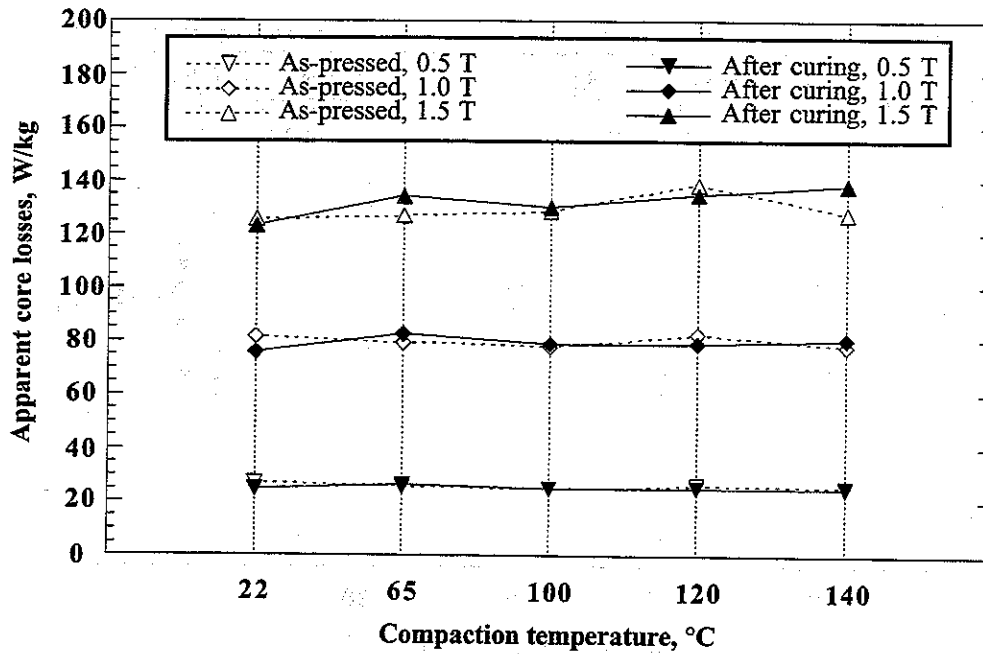
The effect of the frequency on specific losses calculated according to equation (1) for rings pressed at different temperatures is shown in Figure 12. At low frequency levels (1 kHz), where the total loss is dominated by hysteresis loss, specific losses are similar for all materials with a value near  $0.7 \mu\text{W}/\text{cm}^3$ . At frequencies higher than approximately 10 kHz, specific losses of materials varies with the compaction temperatures: lowest, medium and highest losses are respectively obtained for rings pressed at room temperature, 120 to 140°C and 65 to 100°C. Materials with the lowest losses correspond to those with the highest resistivities. However, it is useful here to recall that these materials are designed for applications at low and medium frequency. Nevertheless, by using warm-pressing it is possible to maintain low specific losses. For instance at 10 kHz, specific losses of 13 and  $16 \mu\text{W}/\text{cm}^3$  were measured on rings pressed at ambient (22°C) and 120°C respectively after curing at 175°C during 1 hour.



(a) 60 Hz

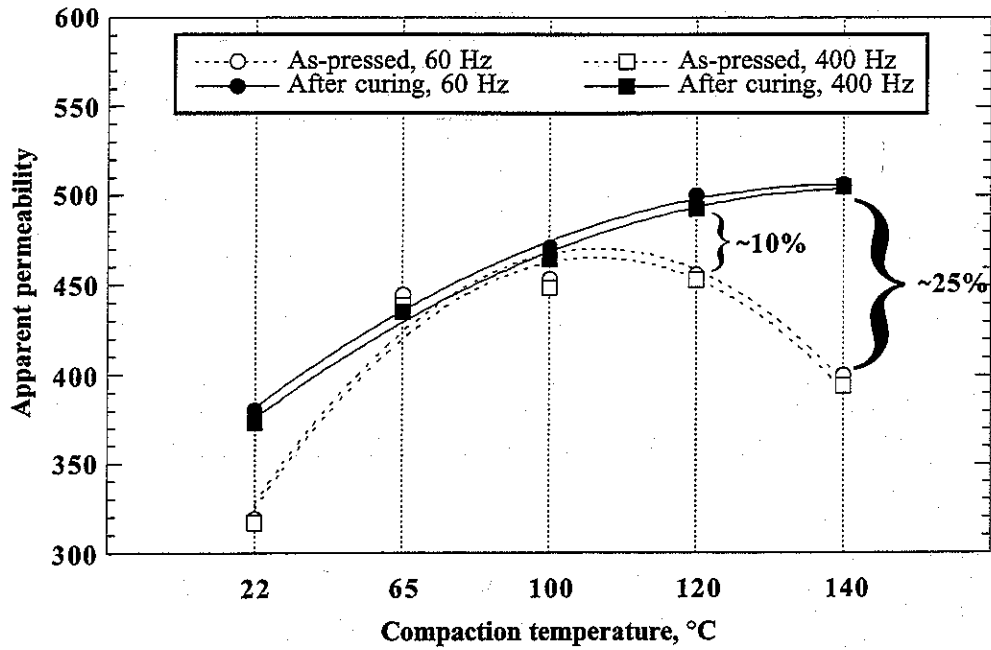


(b) 400 Hz



Figures 9: Effect of compaction temperature on apparent core losses of rings pressed at 620 MPa (45 tsi): a) 60 Hz and b) 400 Hz (As-pressed or after curing at 175°C for one hour)

(a) 0.5 T



(b) 1.0 T

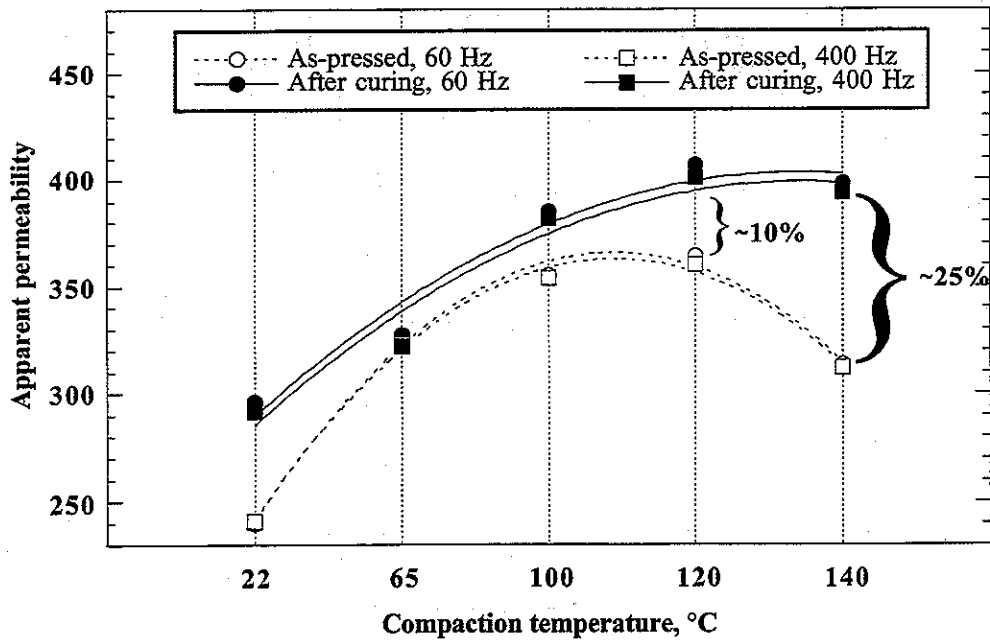
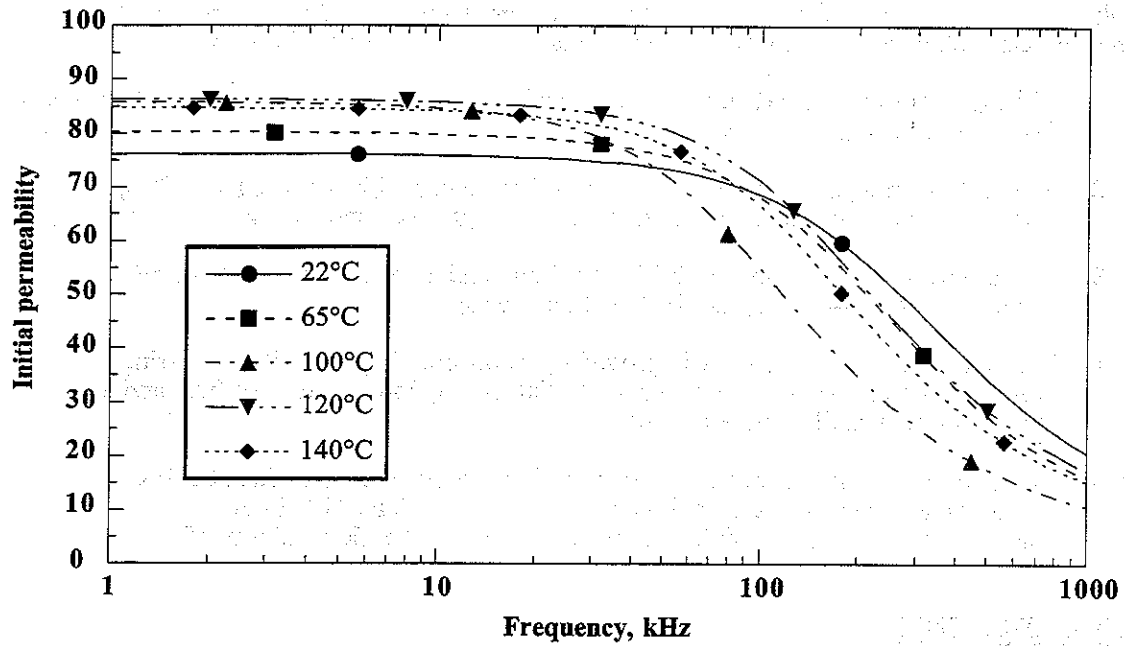
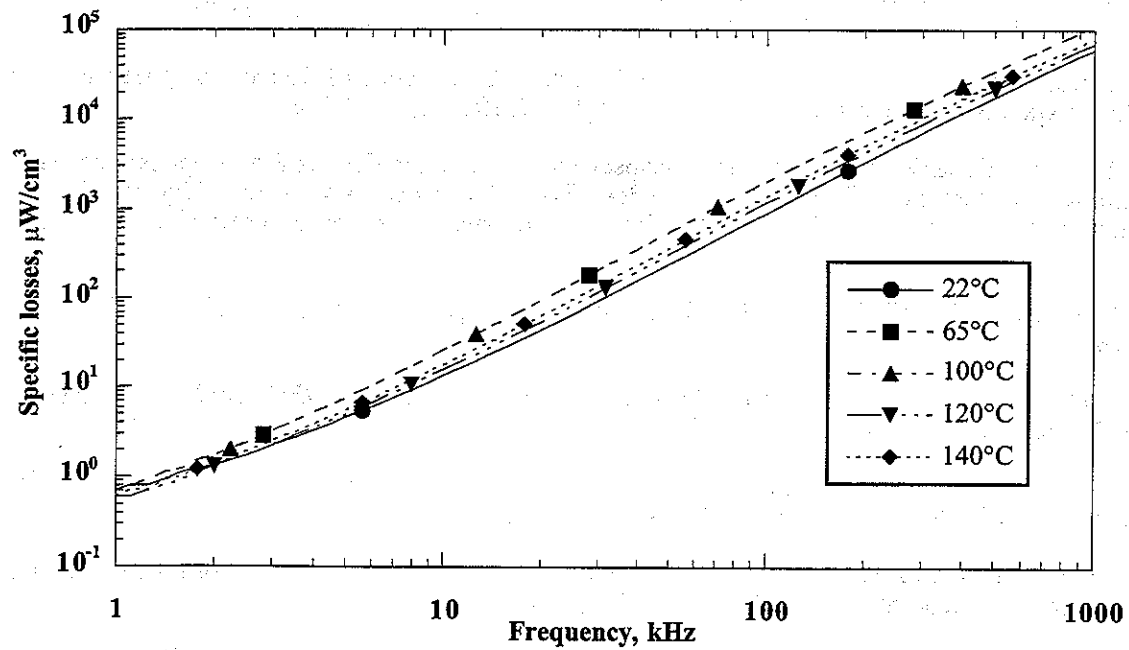


Figure 10: Effect of compaction temperature on apparent permeability at 60 and 400 Hz of rings pressed at 620 MPa (45 tsi): a) 0.5 T and b) 1.0 T. (As-pressed or after curing at 175°C for one hour)



**Figure 11: Effect of frequency on initial permeability of rings pressed at 620 MPa (45 tsi). (After curing at 175°C for 1 hour).**



**Figure 12: Effect of frequency on specific losses @ 0.001 T (10 G) of rings pressed at 620 MPa (45 tsi). (After curing at 175°C for one hour)**

#### 4. CONCLUSIONS

In this paper it has been demonstrated that warm-pressing of iron-resin composite powders can broaden the range of physical, mechanical and both DC and AC magnetic properties. Depending on the type of application, the needed properties can be obtained by varying the compaction temperature, with or without a curing treatment at 175°C for 1 hour in air. These composite mixes are specifically designed for AC applications at low and medium frequency but quite impressive DC and AC properties at high frequency are also achieved. The following conclusions can be drawn from this study:

- 1- The highest resistivities (250  $\mu\Omega\text{-m}$ ) are obtained by pressing at room temperature (22°C) and curing;
- 2- The highest strength (130 MPa or 18,850 psi) are obtained by pressing at 65°C (temperature usually reached during industrial production) and curing;
- 3- A combination of high density (7.33 g/cm<sup>3</sup>) and high strength (60 MPa) can be achieved by warm-pressing at 120°C without curing;
- 4- DC magnetic properties can be improved by warm-pressing at 120°C and curing: close to 1.6 T of magnetic induction at (19900 A/m or 250 Oe) applied field and a maximum permeability close to 300;
- 5- Improved AC magnetic properties are also obtained by warm-pressing at 120°C and curing: an apparent AC permeability and core loss at 400 Hz (1.0 T) of approximately 400 and 80 W/kg respectively and an initial permeability of 86.

#### ACKNOWLEDGMENTS

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#### REFERENCES

- [1] B. Weglinski, "Soft Magnetic Powder Composites - Dielectromagnetics and Magnetodielectrics", *Reviews on Powder Metallurgy and Physical Ceramics*, Vol. 4, No. 2, 1990, pp. 79-154.
- [2] A. Kordecki and B. Weglinski, "Theoretical Aspects of Compaction of Dielectromagnetic Powder Metallurgy Materials", *Powder Metallurgy*, Vol. 13, No. 2, 1988, pp. 113-116.
- [3] D. E. Gay, "Higher Performance Microencapsulated Powders for Various P/M Applications, 1995 International Conference & Exhibition Powder Metallurgy & Particulate Materials, May 15-17, Vol. 3, Compiled by M. Phillips and J. Porter, Metal Powder Industries Federation, Princeton NJ, 1995, p. 11-103.
- [4] T.A. Soileau and L. W. Speaker, "Powdered Iron Core Magnetic Devices", U.S. Patent No. 4,601,765.
- [5] St. Kubzdela and B. Weglinski, *Soft Magnetic Powder Composites and their Applications*, Metal Powder Reports, Vol. 37, No.1, 1982, pp. 21-23.
- [6] A. Kordecki, B. Weglinski and J. Kaczmar, *Properties and Applications of Soft Magnetic Powder Composites*, Powder Metallurgy, Vol.25, No. 4, 1982, pp. 201-208.
- [7] M. Huang, H. Debruzzi and T. Riso., "Stator and method of constructing same for high power density electric motors and generator", U.S. Patents # 5, 382, 859.
- [8] F. Chagnon, C. Gélinas and Y. Trudel, "Development of high density materials for P/M applications" 1994 International Conf. & Exhibition on Powder Metallurgy & Particulate Materials, May 8-11, Metal Powder Industries Federation, Princeton NJ, Metal Powder Industries Federation, APMI, Toronto, 1994.

[9] C. Gélinas, "Consolidation of iron-resin composites by warm-pressing without curing", QMP confidential report, File 8-36402.

[10] C. Lall, *Soft Magnetism-Fundamentals for Powder Metallurgy and Metal Injection Molding*, Monographs in P/M Series No.2, Metal Powder Industries Federation, Princeton, NJ, 1992, p. 141.

[11] C. Heck, *Magnetic Materials and their applications*, 1974, Crane, Russak & Company, Inc., 1974, p. 770.

[12] S. Pelletier, C. Gélinas, *Composites à base de poudre de fer pour la production de pièces magnétiques douces*, IMI Confidential report, IMI95RT-60446-63885-C

[13] P. Jansson and M. Persson, *Composites Pave the Way to the Electrical Machine Designs of the Future*, Euro PM'95, Birmingham, UK, 1995.