

DEVELOPMENT OF AN IRON-RESIN COMPOSITE MATERIAL FOR SOFT MAGNETIC APPLICATIONS.

C. Gélinas, F. Chagnon and S. Pelletier*.

**Quebec Metal Powders Limited
Tracy, Québec, Canada J3R 4R4**

***Industrial Materials Institute
National Research Council Canada
Boucherville, Québec, Canada J4B 6Y4**

ABSTRACT

Soft magnetic materials produced by the P/M sintering technique have proven their advantage and usefulness and are today commonly used for DC applications. On the other hand, many applications are not pure DC but rather experience alternating magnetic fluxes inducing eddy currents, e.g., rotating devices such as brushless DC motors. For these types of applications, lamination stacks are usually preferred. However, the development of composite powder materials in which the individual pure iron particles are insulated from each other opens new possibilities. Emerging industrial interests for using these composite materials are based on economical advantages and design flexibility that enables the production of complex parts which cannot be made with traditional stack laminations.

In this paper, the effect of the manufacturing technique (dry or wet mixing) and the use of a lubricant on the mechanical and AC magnetic properties at low and high frequencies are evaluated and discussed. A new iron-resin composite material for low frequency applications is presented and its properties are compared with those of lamination stacks.

INTRODUCTION

Soft magnetic powder composites, often referred to as dielectromagnetics, are ferromagnetic materials (usually iron powders) and dielectrics such as inorganics, thermoplastics or thermosets. Dielectromagnetics in which iron particles are insulated from each other with dielectrics are specifically designed for applications in alternating magnetic fields. Historically, magnetic cores made of dielectromagnetics and electrical sheets appeared almost at the same time in the early 1880's. The former were proposed by Fritts while laminated thin steel sheets were developed by Edison. Stack laminations were rapidly used in medium power electrical motors while composites found applications in radio engineering and telecommunication. With the introduction of the soft magnetic ferrites into the magnetic materials market, research and production of magnetic cores of dielectromagnetics were discontinued. However, electrical sheets and ferrites do not cover the full range of technical requirements associated with these applications. The gap can be undoubtedly filled by dielectromagnetics, as illustrated in Figure 1 proposed by Weglinski [1].

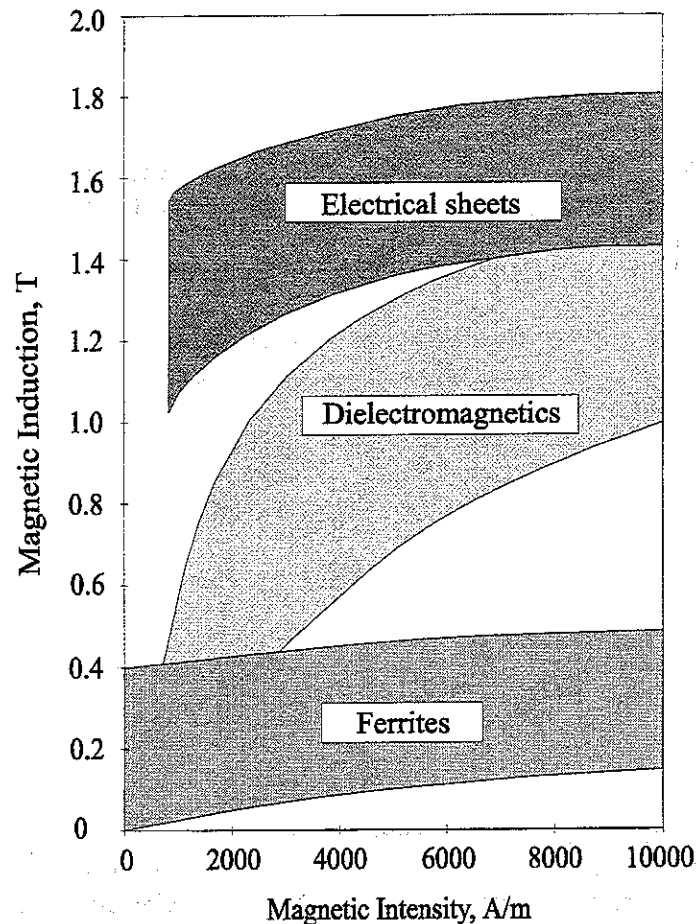


Figure 1. Magnetization characteristics of electrical sheets, dielectromagnetics and soft ferrites [1].

With the continuous developments in P/M processes and the formulation of new polymeric materials, new magnetic materials and applications become feasible. For instance, up to now, the design of electrical machines has been dictated by the constraining anisotropic properties of laminations. Dielectromagnetics with isotropic and improved properties open doors to better adapted designs for existing and new applications.

This paper describes a unique composite material, ATOMET EM-1, designed for AC magnetic applications at low and medium frequencies. Materials and processing aspects related to the design and development of a new dielectromagnetic material are discussed. Physical, mechanical and magnetic properties are presented.

INPUT DESIGN VARIABLES

Literature review and market studies indicate that there is a need for materials capable of filling the gap between properties of ferrites and those of steel sheets. Because of their large utilization and their properties, steel sheets appear as ideal benchmark products for dielectromagnetics. Before developing a new product for AC magnetic applications, some input design variables have to be taken into consideration such as materials, processing, customer needs and obviously a good understanding of the property-application relationship.

Magnetic properties.

Magnetic properties change when subjected to alternating magnetic fields, especially relative permeability and core losses. As well described by Oliver and Rutz [2], the two main components of core losses, namely hysteresis and eddy current losses, are functions of frequency. Hysteresis loss increases with the first power of frequency and dominates at low frequency while eddy current loss is a function of the second power of frequency and dominates at high frequency. Eddy currents also affect the measured permeability since they induce an opposing magnetic field in the material lowering the measured induction level and consequently the measured permeability. It is thus important to keep eddy currents as low as possible. Laminations, due to their insulation between sheets, reduce eddy currents but are anisotropic in nature and are not effective when magnetic fluxes travel parallel to their plane. On the other hand, in dielectromagnetics made from insulated iron particles, eddy currents are restricted from flowing between iron particles and are thus isotropic in nature.

Materials.

The purity, particle size and particle shape of the base iron powder are key factors in improving magnetic properties [1]. Impurities such as carbon, oxygen, sulfur and nitrogen decrease powder compressibility. In addition, they can distort the lattice of the crystal structure and interfere with movement of the magnetic domains [3]. This increases hysteresis loss which is the main part of the total apparent losses at low frequencies [4]. Powder particle size also affects magnetic properties. For instance, coarse particles are preferable for low frequency applications where high permeability is needed while for high frequency applications, fine particles are preferred in order to reduce eddy current losses. Powder particle shape is also important: spherical or granular shape particles with low open porosity are suitable since insulation of iron particles can be achieved with a minimum amount of dielectric.

The dielectric basically has two functions: insulate iron particles and provide adequate mechanical strength [1]. Engineering thermoplastics and thermosets fulfill these requirements and are generally used as organic dielectrics. Thermoplastics have to be heated above their melting or softening point during shaping while thermosets can be processed at room temperature followed by a curing treatment at relatively low temperature. However, if a high temperature heat treatment has to be carried out, inorganic dielectrics such as oxides, phosphates or glasses are more suitable. In some cases, these two types of dielectrics can be combined to improve the insulation between iron particles and maintain good strength. These are referred to as doubly-coated composite powders. It is worth mentioning that the modulus-temperature behavior of a thermoset is similar to that of a thermoplastic at low temperature, but at high temperature, the modulus of a thermoplastic decreases continuously while that of a thermoset is constant [5].

Processing.

Different techniques exist to manufacture single-coated dielectromagnetic powders, the objective always being to insulate iron particles from each other with a dielectric. For instance, this can be achieved by conventional blending of solid constituents or by wet blending the iron with the insulating material. This latter process can also be made according to many variants: the insulating material can be melted and sprayed on the iron particles or a solution carrying the insulating material, dissolved or in emulsion, can be sprayed on the iron particles.

Customer needs.

From the customer point of view, low cost products are needed that translate into simple processing for the powder manufacturer. Core loss is obviously a major concern and a new dielectromagnetic material has to perform as well as or better than a lamination. For the first phase of a development program, 1008 steel sheet laminations currently used in low frequency AC applications were selected as benchmark products.

PRODUCT DEVELOPMENT PROGRAM

Materials.

ATOMET 1001HP, a high purity and high compressibility water-atomized iron powder, was selected as the ferromagnetic component of these dielectromagnetics. A scanning electron micrograph of this iron powder is presented in Figure 2.

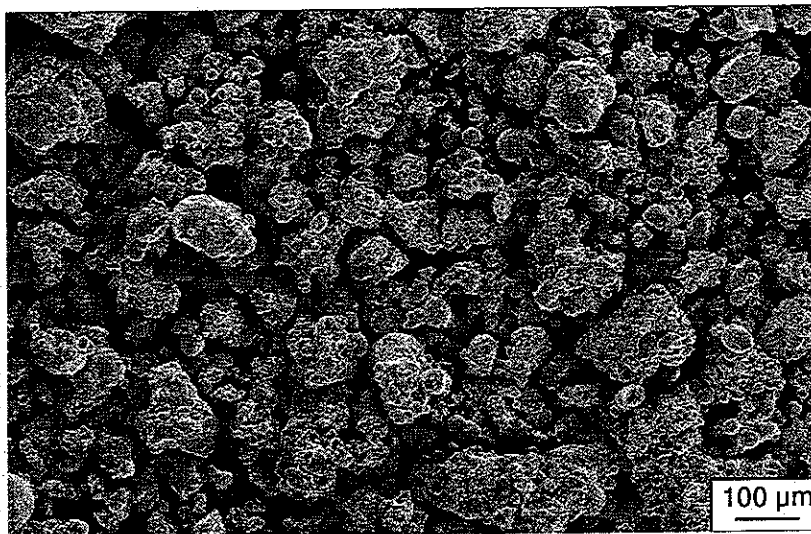


Figure 2. Particles of the ATOMET 1001HP iron powder used as the ferromagnetic component in the manufacturing of dielectromagnetics.

The particles are irregular in shape and have very little open porosity, two characteristics that allow a small amount of resin to efficiently insulate the particles. A solid thermoset resin was also selected because of the simple processing used for shaping: compaction followed by curing at low temperature. Moreover, there was a concern for mechanical properties of end-products that would not be affected by temperature. On that aspect, thermosets perform better than thermoplastics and for certain applications running at relatively high temperatures, it is an important issue. Preliminary experiments were designed to test a large variety of epoxy and phenolic resins. Based on mechanical and magnetic performances, a unique phenolic thermoset resin was finally selected.

Processing.

In order to keep the cost as low as possible a conventional dry blending process was tested. This was possible because of the type of insulating material used. Indeed, since the thermoset resin showed a very good capability to wet the iron particles during the curing treatment, it was not necessary to uniformly coat the iron particles. Consolidation of parts was done in the laboratory in conditions very close to those of production. For instance, the pressing temperature was set at 65°C to reflect temperatures normally reached during production runs on compacting presses. Parts were cured in air at 175°C for one hour. This simple processing route was compared with more sophisticated routes where iron particles were, in the first case, wetted or, in the second case, encapsulated with the thermoset resin using a fluidized bed processor. In the former process, a liquid resin was sprayed on the iron particles in a V-type blender/dryer, the mix was homogenized and then vacuum dried. In the latter, a fluidized bed coating processor was used

to coat the iron particles. This apparatus has a coating chamber located at the bottom where the iron powder is sprayed with the resin solution and an expansion chamber at the top where the solvent evaporates leaving a uniform resin layer on iron particles.

Characterization.

The apparent density and Hall flow rate of the powder mixes were measured in accordance with MPIF's standards 03 and 04 respectively. Electrical resistivity was measured on TRS bars before and after curing using a four-point method. Five measurements were made on both sides of the bars (top and bottom) and averaged. Transverse rupture strength was determined according to MPIF's standard 41. Magnetic characterization (DC and AC at low and high frequency) was carried out on rings pressed at 65°C/45 tsi (620 MPa) and cured in air at 175°C for 1 hour. Dimensions of the rings were 4.38 x 5.27 x 0.62 cm (1.72" x 2.07" x 0.25") with an inner to outer diameter ratio of 0.83. DC and AC low frequency measurements were carried out using a SMT/ACT-500 magnetic hysteresis graph. For the DC magnetic characterization, rings were wound with 600 primary turns of #24 gauge insulated copper wire and 150 secondary turns of #30 gauge insulated copper wire. For the AC characterization at low and medium frequency, rings were wound with 250 primary turns of #24 gauge insulated copper wire and 250 secondary turns of #30 gauge copper wire. A reference lamination ring with similar dimensions was also tested for its DC and AC low frequency properties. This lamination ring was made out of seven 0.032" thick 1008 steel sheets.

The iron-resin composite material was also evaluated at high frequency from 1 kHz to 1 MHz. The initial permeability and the loss factor "tan δ " were measured using a HP 4192A LF impedance analyzer connected to the coil with a HP 16047A test fixture. An excitation of 1 V_{rms} producing a field B < 5x10⁻⁴ T (5 Gauss) was applied. In that case, rings were wound with 96 turns of #24 gauge insulated copper wire.

RESULTS AND DISCUSSION

Physical and mechanical properties.

It is important to those in the P/M manufacturing industry that mixes can be processed easily. The apparent density, flow and compressibility of the new composite powder mix meet that requirement. Typical apparent density and flow rate of the developed iron-resin mixes are 2.75 g/cm³ and 28 s/50 g respectively. The compressibility of the composite mix has been evaluated by pressing TRS bars. Since there is no difference in density between green and cured bars, only densities after curing at 175°C for 1 hour are presented. Results are shown in Figure 3 for two compacting temperatures: 22°C or room temperature and 65°C. The room temperature plot reflects results usually obtained on a laboratory press while the 65°C plot reflects results obtained on a production press where a slight temperature increase normally occurs during compaction. There is an increase in the density of the bars with an increase of the compacting temperature from 22°C to 65°C. This is especially true for compacting pressures lower than 50 tsi (690 MPa).

These iron-resin composites have excellent mechanical strength after curing. The strength of iron-resin composite specimens is given in Figure 4 for two compacting temperatures: 22°C and 65°C. After curing, values close to 20,000 psi (140 MPa) can be reached by pressing at 65°C in the 40-60 tsi (550-825 MPa) compacting pressure range. At room temperature, strength increases continuously with the compacting pressure. In fact, the strength is closely related to the density reached during compaction. This is illustrated in Figure 5 where the strength increases with the density for the two compacting temperatures. However, for a given density, strength values are slightly higher for parts pressed at 65°C. Before curing, it has been verified that strength is much lower. For example, green strength values of about 3400 psi (23 MPa) are obtained at a compacting pressure of 45 tsi (620 MPa).

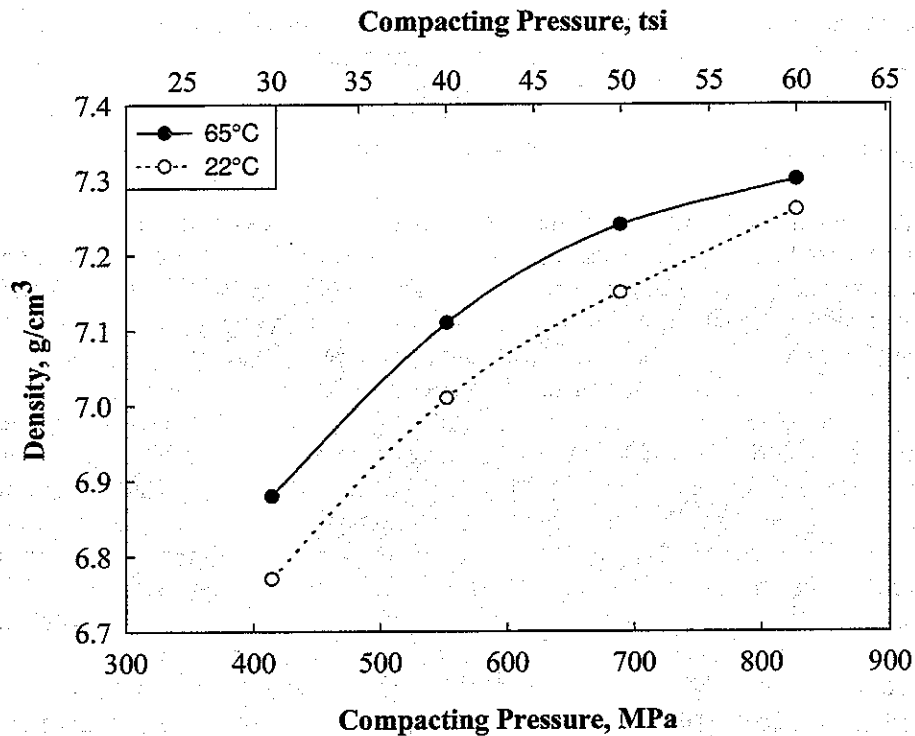


Figure 3. Effect of compacting pressure on density of the iron-resin composite pressed at room temperature or 65°C and cured in air at 175°C for one hour.

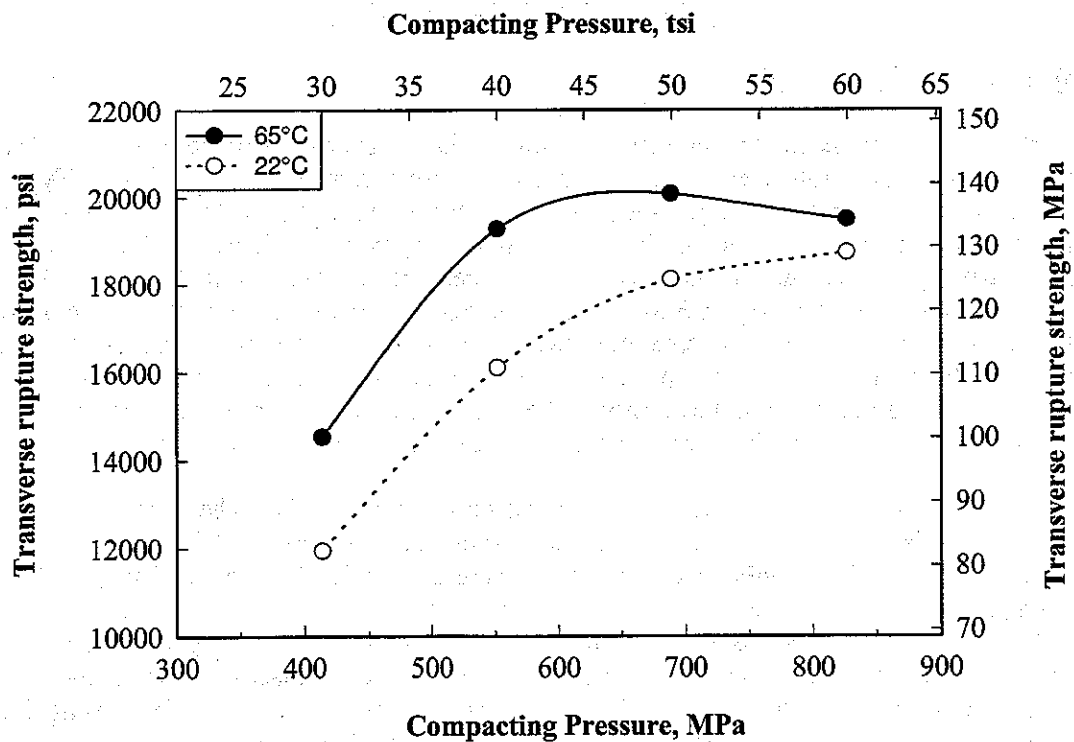


Figure 4. Effect of compacting pressure on strength of the iron-resin composite pressed at room temperature or 65°C and cured in air at 175°C for one hour.

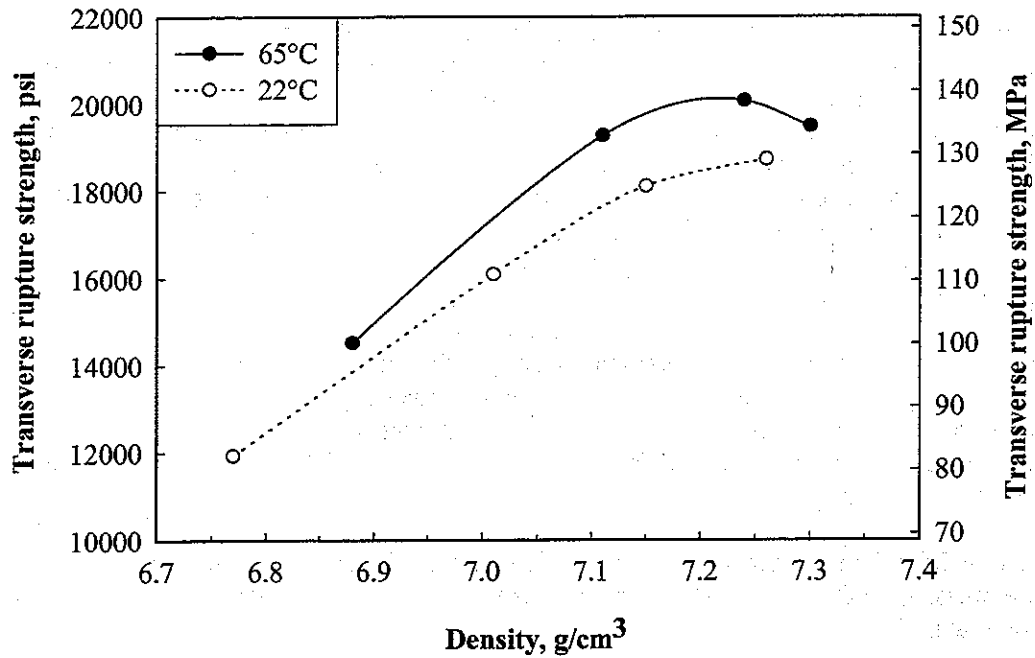


Figure 5. Effect of density on strength of the iron-resin composite pressed at room temperature or 65°C and cured in air at 175°C for one hour.

Another useful material property for magnetic applications in an alternating field is the electrical resistivity. This property gives an indication of the degree of insulation between iron particles, which insulation governs losses by eddy currents. The effect of compacting pressure on resistivity of TRS bars, before and after curing, is illustrated in Figure 6.

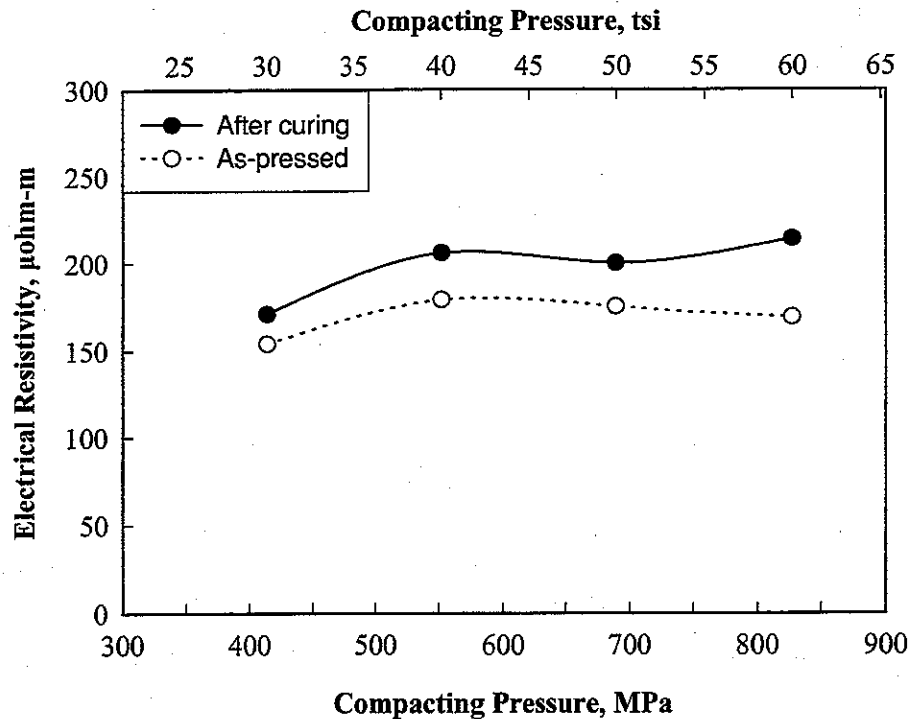


Figure 6. Effect of compacting pressure on electrical resistivity of the iron-resin composite pressed at 65°C (before and after curing in air at 175°C for one hour).

The compacting pressure has little effect on electrical resistivity of bars pressed at 65°C. On the other hand, curing results in an increase of about 15% in resistivity compared with as-pressed bars. This is likely due to a change in resin resistivity during curing. Values of about 200 $\mu\text{ohm}\cdot\text{m}$ obtained with these iron-resin composites pressed at 65°C are much higher than those achieved with most of the soft magnetic materials used at low frequency. For instance, resistivity for low carbon steels and Fe-Si steels (oriented or non-oriented) is around 0.1 and 0.5 $\mu\text{ohm}\cdot\text{m}$ respectively. Moreover, contrary to laminated steels, the electrical resistivity of iron-resin composites is really isotropic.

DC magnetic properties.

Iron-resin composite materials or dielectromagnetics are designed for AC magnetic applications. However, the DC magnetic performances are useful for selecting materials and for modeling; the latter becoming more and more necessary. Indeed, it permits one to virtually test materials in real applications in a very short time, reducing prototyping phases (cost and time) and taking full advantage of the isotropic properties of dielectromagnetics.

First quadrant DC magnetization loops at an applied field of 250 Oe (19900 A/m) for the iron-resin composite pressed at 65°C/45 tsi (625 MPa) and cured at 175°C for one hour and for a 1008 steel sheet lamination materials are shown in Figure 7.

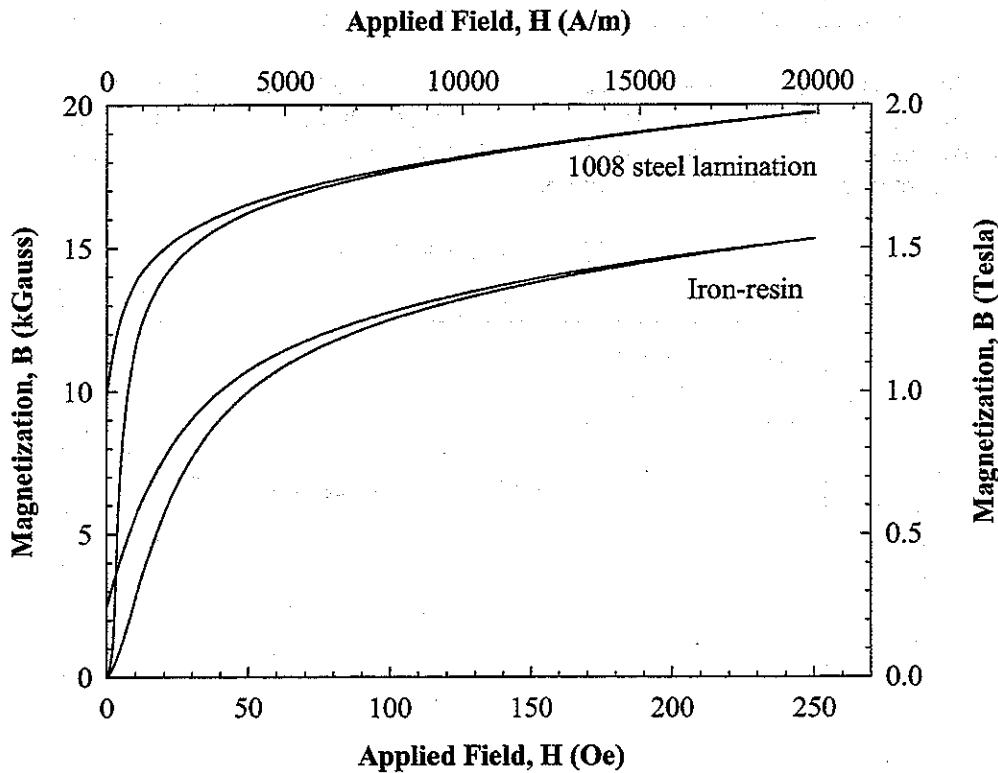


Figure 7. First quadrant DC magnetization loops for the iron-resin composite and a 1008 steel lamination in a 250 Oe applied field (19900 A/m).

The hysteresis loop of the 1008 steel lamination is rather square compared with that of the iron-resin material which is sheared. The shearing is caused by the important air gap distribution in the magnetic circuit [6]. Maximum permeability values reflect these loop shapes: 290 and 1530 for iron-resin composite and lamination, respectively. However, high saturation magnetization at 250 Oe (19900 A/m) applied field is still achieved with the iron-resin composite: 15.3 kG compared with 19.7 kG for the lamination. The

coercive field H_c for the lamination and the iron-resin material is 5.3 and 3.2 Oe (422 and 255 A/m) respectively while the remanent magnetization B_r is 2.5 and 10 kG respectively.

AC magnetic properties.

Apparent core loss and relative permeability at frequencies up to 1000 Hz were also measured for the iron-resin composite material and a reference 1008 steel sheet lamination. Relative permeabilities (B/H) at different frequencies for the iron-resin composite and the reference 1008 steel sheet lamination are given in Figure 8. The permeability of the composite material (Figure 8a) increases with the magnetization, reaches a maximum value at about 5 kG and then decreases. Permeabilities of the lamination (Figure 8b) are higher than those of the composite material but are more affected by the frequency. Indeed, for the lamination, permeability decreases drastically with an increase of the frequency contrary to the composite material which shows a frequency-independant permeability.

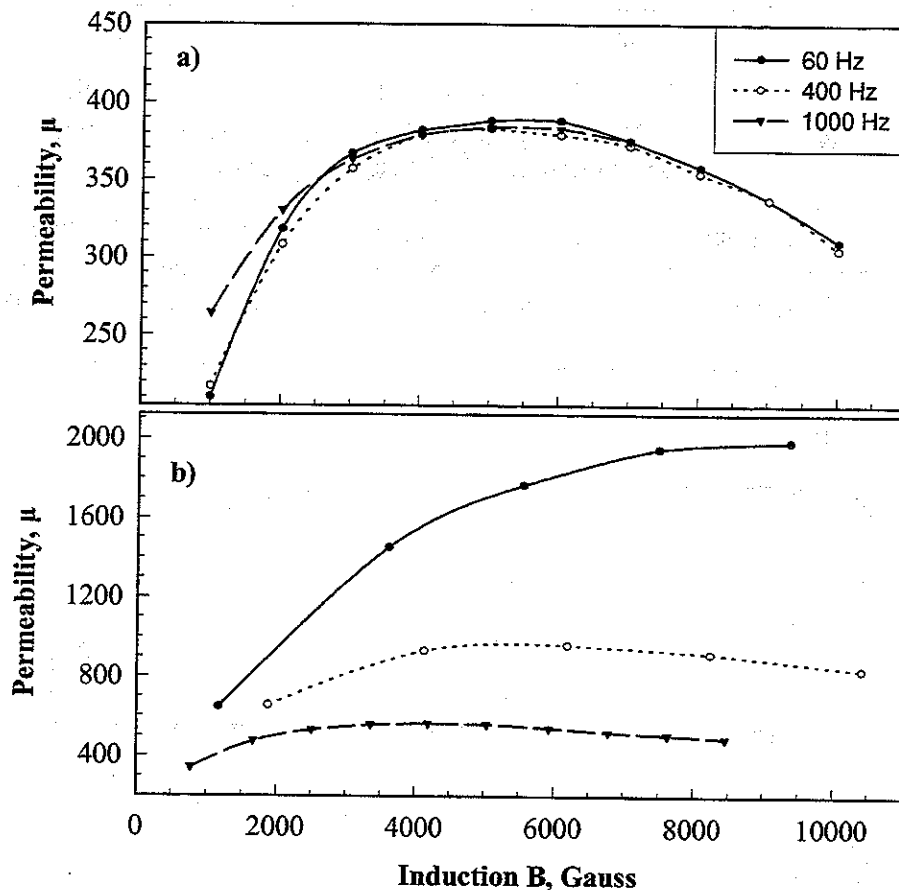


Figure 8. Effect of magnetic induction on relative AC permeability at 60, 400 and 1000 Hz: a) iron-resin composite pressed at 65°C/45 tsi (625 MPa) and cured in air at 175°C for 1 hour; b) 1008 steel lamination.

The effects of frequency on apparent core losses of both the iron-resin pressed at 65°C/45 tsi (620 MPa) and cured at 175°C for one hour and the 1008 steel lamination are shown in Figure 9 at 5 and 10 kG induction levels. There is a cross-over between the two materials: losses of iron-resin composites are lower than those of a lamination at frequencies higher than about 100 Hz. Composites tend to perform better than laminations when frequency and magnetization increase.

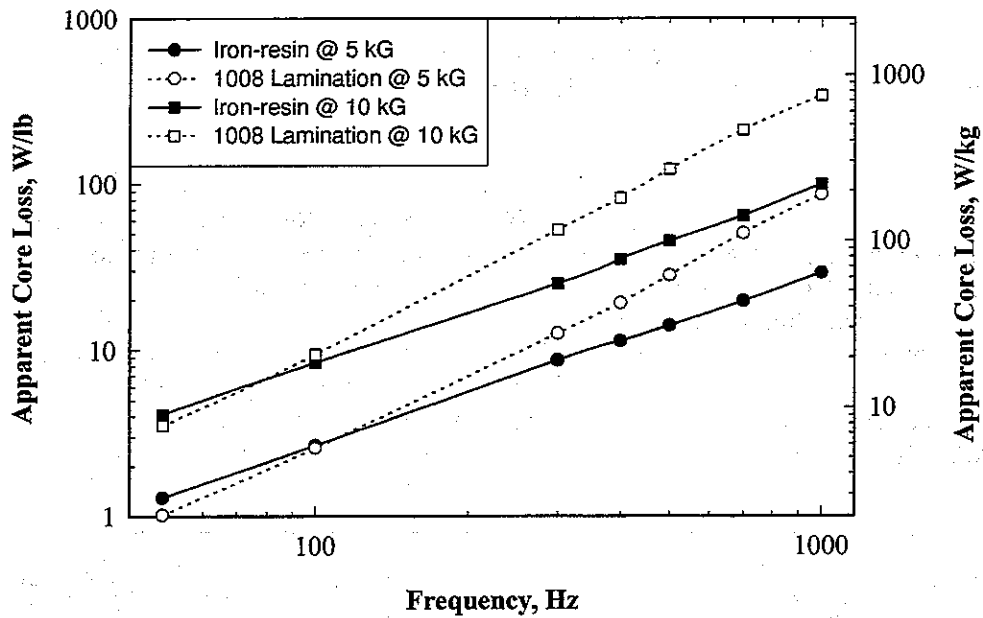


Figure 9. Effect of frequency on core loss of the iron-resin composite and a reference 1008 steel lamination.

Even though the iron-resin system is designed for low and medium frequency applications, the initial permeability and the loss factor $\tan \delta$ or μ''/μ' have also been measured at high frequencies. The variation of these properties with frequency levels ranging from 100 Hz to 1 MHz is illustrated in Figure 10.

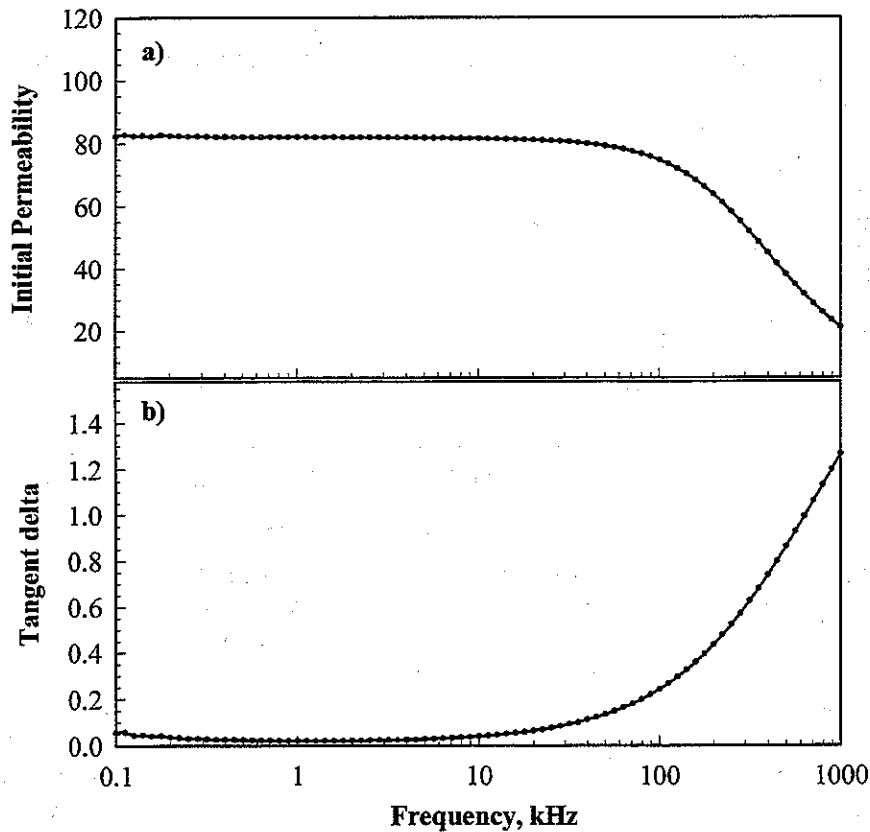


Figure 10. Magnetic properties at high frequencies of iron-resin composites pressed at 65°C/45 tsi (620 MPa) and cured in air at 175°C for one hour: a) initial permeability and b) loss factor $\tan \delta$.

A constant initial permeability between 80 and 83 is measured up to about 40 kHz, after which permeability drops due to an increase of eddy currents. Values of $\tan \delta$ ranging from 0.02 to 0.06 are measured for frequencies up to about 20 kHz. At higher frequencies, values of $\tan \delta$ increase drastically indicating that the insulation between iron particles is no more adequate for such high frequencies, thus resulting in an increase of eddy currents.

Effects of materials processing.

Iron-resin composite mixes made according to three coating procedures, namely conventional dry blending, wet blending and fluid-bed encapsulation were compared. Core loss was measured on rings pressed at 65°C/45 tsi (620 MPa) and cured at 175°C for 1 hour. The variation of core loss with magnetic induction at 60 and 400 Hz is shown in Figure 11 for these three powder processing techniques. There is no significant differences in core loss at these frequencies. Rings pressed from iron-resin mixes made from conventional dry blending perform as well as those made from wet blending or fluid-bed encapsulation.

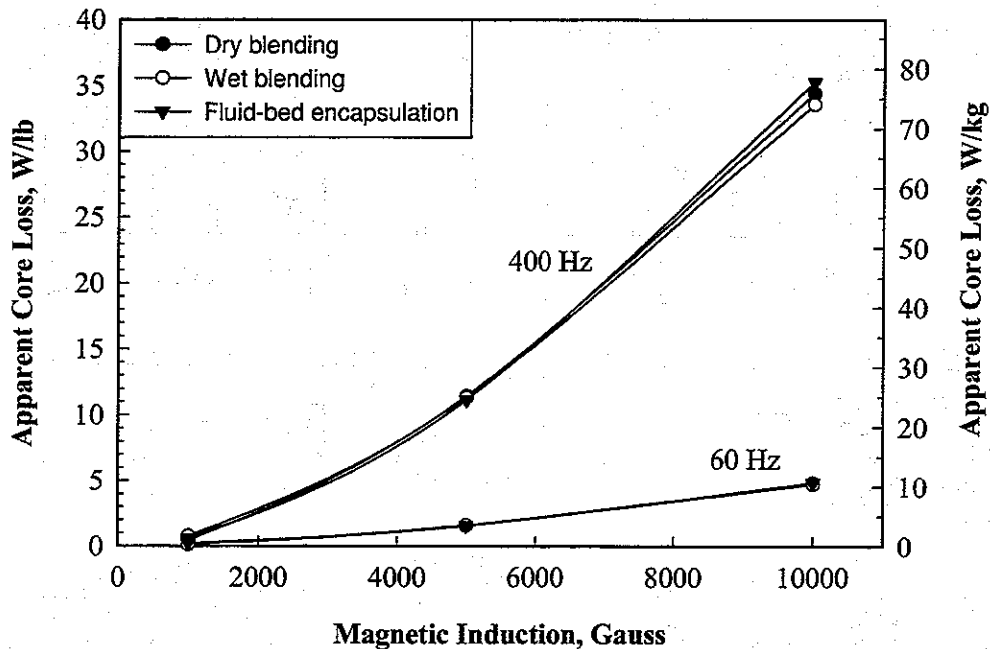


Figure 11. Effect of powder processing on core loss at 60 and 400 Hz (rings pressed at 65°C/45 tsi (620 MPa) and cured in air at 175°C for one hour).

Effects of adding a lubricant to the iron-resin mix.

It is also possible to use a lubricant with iron-resin composite mixes. Because of the curing treatment it is suitable to use a high melting point lubricant such as teflon (polytetrafluoroethylene or PTFE) which is a good insulating material that will not melt during curing. Three lubrication techniques were evaluated: die wall lubrication using a graphite spray, no lubrication and 0.3 wt% teflon admixed in the iron-resin powder. Rings and bars were pressed at 65°C/45 tsi (620 MPa) and cured in air at 175°C for one hour. The density, electrical resistivity, ejection pressure and strength were measured on bars while apparent permeability and core loss were measured on rings at 60 and 400 Hz for a magnetization of 10 kG. The ejection pressure was measured by recording the load necessary to initiate movement of the transverse rupture bar and dividing that load by the area of the bar in contact with the die walls.

The effect of the lubrication technique on physical and mechanical properties of composite bars is illustrated in Figure 12. There is a minor effect on the density of the pressed and cured composite bars (Figure 12a): an increase of 0.04 g/cm^3 is seen by using 0.3 wt% teflon in iron-resin mixes instead of die wall lubrication. For ejection pressure measured on transverse rupture bars, the lowest values are obtained by lubricating the die walls. The electrical resistivity is lower for iron-resin mixes containing teflon: approximately one third the value obtained with composite mixes containing no lubricant. This suggests that the resistivity of the thermoset resin is higher than that of teflon. Finally, an important drawback of using lubricant in the composite mixes is the drop in strength (Figure 12d): a loss of approximately 55% compared with composites containing no lubricant.

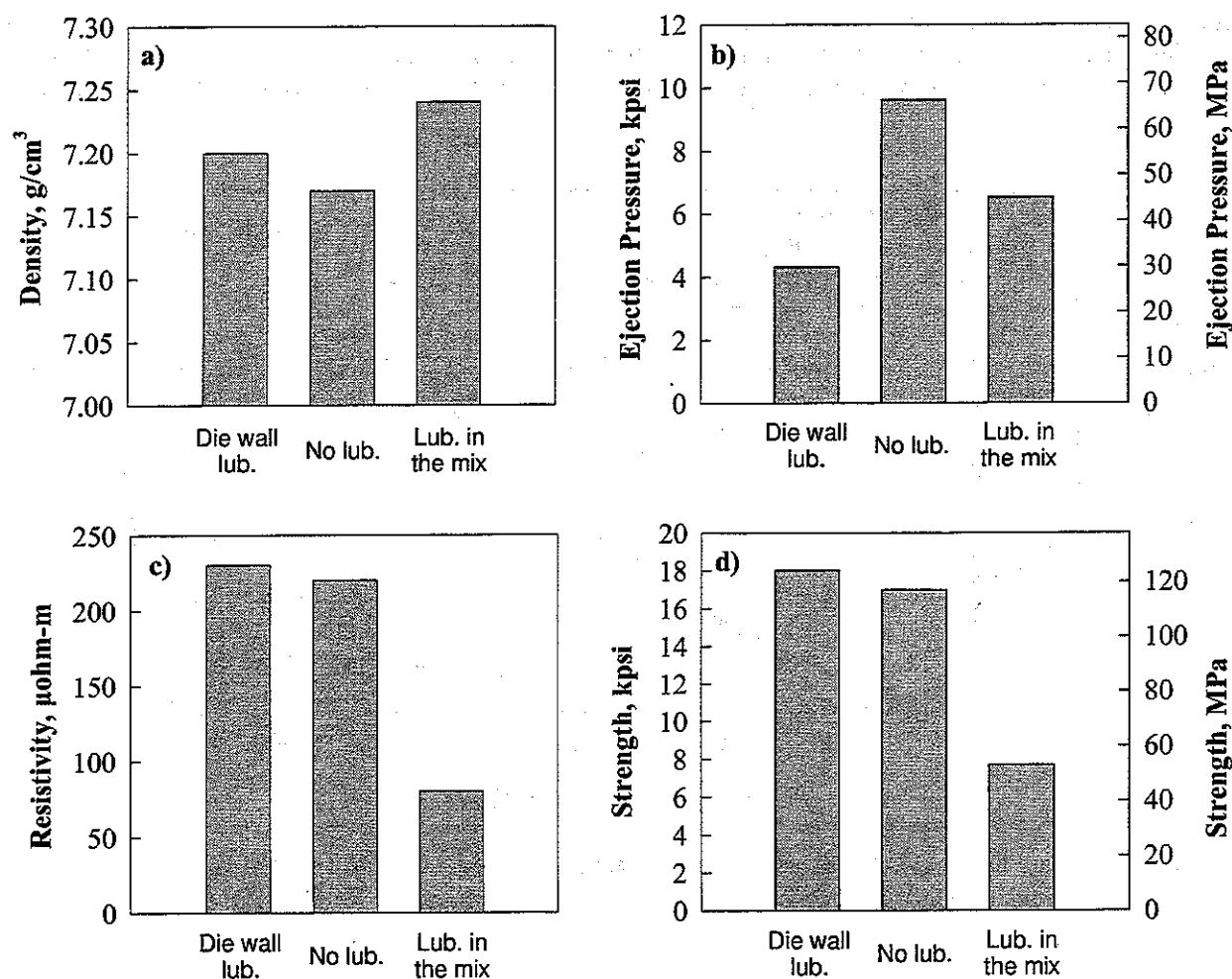


Figure 12. Effect of using die wall lubrication, no lubrication or 0.3 wt% teflon lubricant in the powder mix on properties of iron-resin composite bars pressed at $65^\circ\text{C}/45 \text{ tsi}$ (620 MPa) and cured: a) density; b) ejection pressure; c) electrical resistivity and d) transverse rupture strength.

There is no significant effect of these types of lubrication on the magnetic properties. An average apparent permeability of 320 ± 20 has been measured at 400 Hz and 10 kG of magnetization for the three types of composite rings. The standard deviation is related to the variation in density from one ring to the other. Finally, no difference was measured in core loss at 400 Hz and 10 kG of magnetization: 34.6 W/lb (76 W/kg) was obtained for the three types of rings.

CONCLUSIONS

A low cost high performance iron-resin composite mix has been successfully developed for low and medium frequency applications. The main characteristics of this material system are as follows:

- 1° Ease of shaping by compaction in a wall-lubricated die and curing at 175°C for one hour in air.
- 2° Densities and strengths up to 7.25 g/cm³ and 20,000 psi (140 MPa) respectively are achieved by pressing at 50 tsi (690 MPa) and curing.
- 3° These dielectromagnetics provide isotropic electrical and magnetic properties.
- 4° A DC maximum permeability close to 300 is achieved and a magnetization of 15 kG (1.5 Tesla) is reached at 250 Oe (19900 A/m) applied field.
- 5° Apparent core losses are as good as those usually measured on low carbon steel laminations at 50-60 Hz and are much lower at higher frequencies.
- 6° Apparent core losses as good as those obtained with composites made from wet blended mixes or encapsulated powders are achieved.
- 7° For applications where strength is not a major concern, a lubricant can be added to the iron-resin mix to facilitate pressing without affecting magnetic properties.

REFERENCES

1. B. Weglinski, "Soft Magnetic Powder Composites - Dielectromagnetics and Magnetodielectrics", *Reviews on P/M and Physical Ceramics*, Vol. 4, No. 2, 1990, pp 79-154.
2. C.G. Oliver and H.G. Rutz, "Powder Metallurgy in Electromagnetic Applications", *Advances in Powder Metallurgy & Particulate Materials*, Vol. 3, compiled by M. Phillips and J. Porter, Metal Powder Industries Federation, Princeton, NJ, 1995, pp 11-87 to 11-102.
3. G.Y. Chin, L.L. Harner, M.F. Littmann & J.W. Shilling, "Magnetically Soft Materials", *Metals Handbook Ninth Edition*, Vol. 3, Properties and Selection: Stainless Steels, Tool Materials and Special-Purpose Metals, American Society for Metals, Metals Park, Ohio, 1980, pp 597-614.
4. 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.
5. J.N. Epel, J.M. Margolis, S. Newman and R.B. Seymoir, "Engineering Plastics", *Engineered Materials Handbook*, Vol. 2, ASM International, Metals Park, Ohio, 1988, p. 437.
6. A. Goldman, "The Magnetic Applications Choice Among Ferrite Ceramics, Metallic Strips, or Metal Powder Cores", *JMEPEG*, Vol. 4, 1995, pp 395-400.