EFFECT OF RESIN CONTENT AND IRON POWDER PARTICLE SIZE ON PROPERTIES OF DIELECTROMAGNETICS.

L.-P. Lefebvre, S. Pelletier, B. Champagne and C. Gélinas*

Industrial Materials Institute/National Research Council Canada,
75 de Mortagne, Boucherville, Québec, Canada J4B 6Y4

*** Quebec Metal Powders Limited, 1655 Marie Victorin**
Tracy, Québec, Canada J3R 4R4

ABSTRACT

Powder metallurgy permits the manufacturing of iron-dielectric composites (dielectromagnetics) with high induction, low losses and isotropic properties. Compared to laminated steels, dielectromagnetics made by powder metallurgy show considerably lower eddy current losses and, in comparison with soft ferrites, they have higher magnetic saturation polarization and much better machinability [1]. The resin content and the particle size distribution of the iron powder have important effects on the physical, mechanical and magnetic properties of dielectromagnetics.

This paper presents the effect of resin content and particle size distribution of iron powder on the mechanical, physical and magnetic properties of dielectromagnetics at low and high frequencies.

1. INTRODUCTION

Dielectromagnetics are composite materials consisting of ferromagnetic powder (predominantly iron powder) and a dielectric [2]. Iron powder is mixed with a non magnetic polymer binder to produce distributed air-gap materials whose properties depend on the relative proportions of magnetic powder and binder [3]. Among the interesting features of dielectromagnetics, there are:

- Isotropic magnetic and thermal properties;
- Nearly wasteless production and ease of fabrication;
- Great penetration depth of the electromagnetic wave;
- Low magnetic losses in alternating fields;
- Thermally stable properties;
- Reduction of the fringing flux compared to discrete air-gap cores.

Dielectromagnetic parts can be manufactured using conventional powder metallurgy techniques thus reducing the need to perform additional machining operations [4]. Iron powders for the fabrication of dielectromagnetics should have high compressibility and good magnetic properties such as high permeability and low coercive force. This is achieved by using iron powders with very low impurity content [5]. The dielectric material must have a good capability to bind and insulate the iron particles. The dielectric must also be capable of being applied as a very thin layer, must be mechanically strong at all stages of manufacturing [6] and its amount must be adjusted to optimize the magnetic and mechanical properties.

Permeability, μ, is a measure of the ability to magnetize a material. It is expressed by the ratio B/H, where H represents the magnetizing field strength necessary to produce the magnetic induction B [7]. Permeability depends on the magnetic properties of the starting material and the weight fraction of the magnetic material in the composite. Permeability increases with the filling factor V_{Fe}/V, where V_{Fe} is the iron volume and V the total volume [6]. The maximum core permeability μ_{max} can be expressed as follows [8]:

$$\mu_{\max} = \mu_b^{\frac{V_{Fe}}{V}} \tag{1}$$

The bulk permeability, μ_b, is a derived term which is not identical to μ_{Fe} and is markedly affected by the thickness of the insulating layer between iron particles. The latter is referred to as an "air-gap" because its effect on the magnetic circuit is the same as air. The large number of thin air-gaps between iron particles forms distributed air-gaps having the same effect as a discrete air-gap in the magnetic circuit [3]. The presence of air-gaps in a magnetic circuit shears the hysteresis loop. This is illustrated in Figure 1.

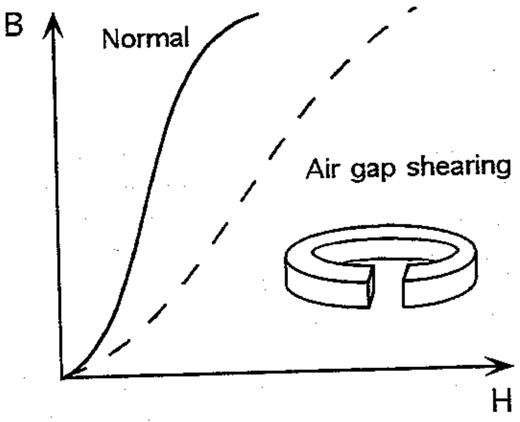


Figure 1: Shearing of a hysteresis loop by application of an air-gap in a magnetic circuit

The slope of the B-H curve is reduced and linearized, and the permeability, though lowered, is made constant when an air-gap is introduced into the magnetic circuit [9]. The effective permeability of a toroid with an air-gap is given by [10]:

$$\mu_e = \frac{\mu}{1 + \mu(l_a / l_m)} \tag{2}$$

where μ is the permeability of the material, l_a the path length of the magnetic field in the air-gap and l_m the path length in the magnetic material. An increase of the air-gap length l_a thus reduces the effective permeability of the material.

The initial permeability, characterizing the behavior of the material at low induction, is structure sensitive and increases as the mean magnetic domain size increases [11]. In isolated particles, the magnetic domain size increases with the particle size, thus the initial permeability of ferromagnetic powder increases when the particle size increases.

Besides the density e.g., filling factor, and the particle size distribution, the particle shape and orientation as well as the core condition (compacted/annealed) also influence the permeability and the shape of the magnetization curve of the composite [12].

When a material is exposed to an alternating magnetic field, it dissipates energy. The power dissipated under an alternating field is defined as the core losses. These losses are composed of hysteresis losses and eddy current losses. Hysteresis losses are due to domain wall movements during cyclical magnetization and can be expressed as follows:

$$P_h = K_h f B^n \tag{3}$$

where K_h is a constant, f is the frequency and B is the magnetic induction. The motion of the domain walls is impeded by pinning centers, e.g., non-magnetic inclusions such as oxides, carbides, and sulfides, and by grain boundaries in the plane of the walls. The density of the pinning centers should be as low as possible in soft magnetic materials used in alternating fields. This means that the ferromagnetic material ideally consists of only one phase, has grains as large as possible and impurity contents as low as possible [8].

When an alternating field is applied to magnetize a ferromagnetic body, an electromagnetic force (emf) is set up in the body. If the material is a good conductor, the induced emf can produce appreciable amounts of currents, called eddy currents, which give rise to an energy loss through Joule (resistance) heating [13]. Aside from the loss of efficiency, the excess heat can lowers the saturation, possibly leading to a chain reaction effect. The equation for core losses due to eddy currents is [9]:

$$P_e = \frac{KB^2 f^2 d^2}{\rho} \tag{4}$$

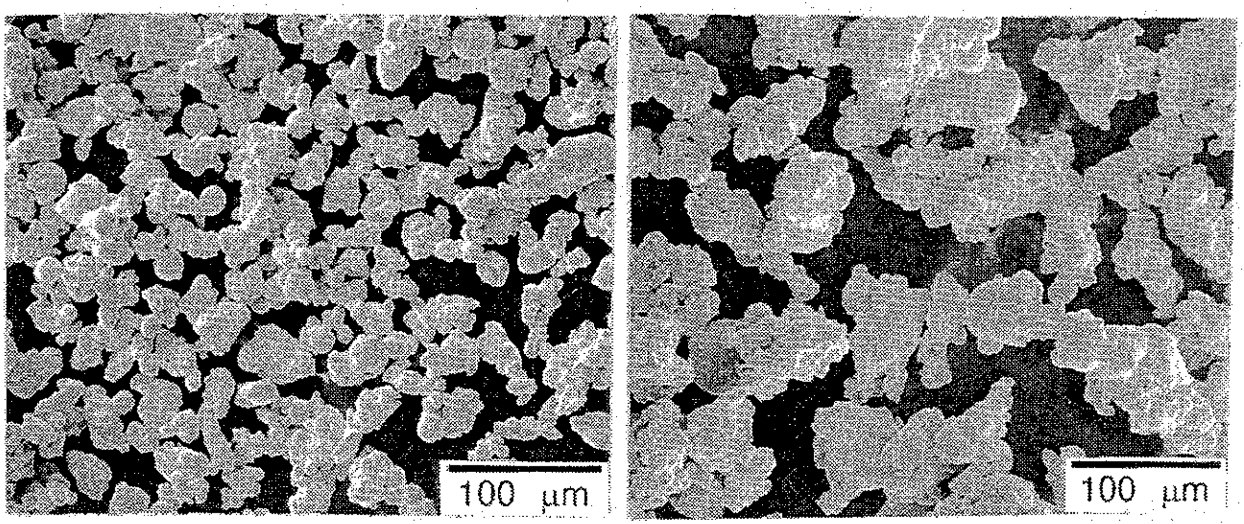
where K is a constant depending on the shape of the sample, B is the magnetic induction, f is the frequency, ρ is the resistivity and d is the shortest dimension perpendicular to the flux path (strip thickness in laminated steels or mean powder diameter in dielectromagnetics). The relative importance of eddy current losses on the total losses increases with frequency since hysteresis and eddy current losses vary respectively with the first and second power of the frequency. Reducing the gage of the strip has been one of the methods of reducing core losses in laminated steels. There are nevertheless cost and technical limitations of reducing the strip thickness [9]. Increasing the amount of dielectric leads to high resistivity and is a method of reducing the losses in dielectromagnetics. Using small particle size distribution of iron is another way to reduce these losses in dielectromagnetics [14]. However, using fine powders and large quantities of dielectric may adversaly affect the permeability. For a given application, these parameters should then be adjusted to optimize the properties.

Applications of dielectromagnetics are conditioned first of all by the required level of magnetic properties of the part in the circuit [15]. The relative importance of the different requirements is determined by the frequency of oscillation of the magnetizing field. At low frequency, high permeability, large saturation magnetization as well as low coercive force are needed to reduce the hysteresis losses. Iron powder with high compressibility is thus preferred for these applications. The dielectric content should be adjusted to obtain an optimum combination of permeability and insulation. At high frequency, the eddy current losses become important and must be minimized. The electrical resistivity and the length of the eddy current path can be tailored by adjusting the dielectric content and the iron powder particle size in order to reduce the eddy current losses.

As outlined above, magnetic properties can be optimized for a particular application by choosing an appropriate particle size distribution of iron powder and the dielectric content. This paper presents the effect of the resin content and the iron particle size on the properties of dielectromagnetics for magnetic applications at low and high frequencies.

2. EXPERIMENTAL PROCEDURE

In these experiments, a high purity water-atomized iron powder suited for magnetic applications (ATOMET 1001HP supplied by QMP) was used. Three particle size distributions were tested: the as-produced powder hereafter called regular powder, the fine powders and the coarse powders. The two latter powders were obtained by screening the regular powder on a 325 Mesh U.S. sieve. The material passing through the sieve was identified as the fine powder (< 45 μm) and the remaining or oversize material was called the coarse powder (> 45 μm). Scanning electron micrographs of the fine and coarse iron powders are presented in Figure 2a and 2b respectively. The regular iron powder is composed of approximately 73 wt% of the coarse powder and 27 wt% of the fine powder.



a) b)

Figure 2: Scanning electron microscope micrographs of a) fine powders (< 45 μm) and b) coarse powders (> 45 μm).

For the evaluation of the effect of the resin content on the physical, mechanical and magnetic properties, mixes were prepared by dry blending the iron powder with different quantities of a thermoset resin powder. For the evaluation of the effect of the iron particle size on the properties, mixes containing 0.8 wt% of the same thermoset resin were dry blended. There was no lubricant in the mixes and die walls were lubricated using zinc stearate spray before each compaction. For each blend, five rectangular bars (3.175 x 1.27 x 0.635 cm) and one ring (o.d. = 5.26 cm, i.d. = 4.34 cm, t = 0.635 cm) were pressed at 620 MPa (45 tsi) in a floating die at room temperature. All the samples were cured at 160°C for 6 hours in air to fully cross-link the resin and increase the mechanical strength of the composite samples.

The density, transverse rupture strength (TRS), resistivity and magnetic properties at low and high frequencies were measured. The density was calculated from the weight and physical dimensions of the parts. Transverse rupture strength tests were made according to MPIF standard 41. The electrical resistivity was evaluated using a four-point contact probe (0.8 cm between contact points) and a micro-ohmmeter adapted for this application. Side and thickness effects were taken into account for the resistivity measurements. Five readings were taken on top and bottom faces of each TRS bar and averaged. The apparent permeability (B/H) and apparent core losses were evaluated at low frequencies (60 Hz and 400 Hz) for a magnetization of 1T using a ACT/SMT-500 computer-automated magnetic hysteresisgraph. Rings were wound with 250 primary turns of 24 gauge copper wire and 250 secondary turns of 30 gauge copper wire. At high frequency, the initial permeability was evaluated from 1 kHz to 1 MHz using a HP4192A LF impedance analyzer equipped with a HP16047A test fixture. The excitation was 1 V_{rms} which produces a field $B < 5x10^{-4}$ T (5 Gauss). Rings were wound with 96 turns of 24 gauge insulated copper wire. The specific losses $p(\omega)$ were evaluated at $1x10^{-3}$ T (10 Gauss) with the following relation:

$$p(\omega) = \frac{1}{2\mu_0}\omega\mu''\left|\frac{B^*(\omega)}{\mu^*(\omega)}\right|^2 \qquad (5)$$

where ω is the frequency (2πf), μ_0 is the free space permeability (4π x 10^{-7} H/m), μ" is the imaginary part of the permeability, $|B^*|$ is the modulus of the induced field and $|\mu^*|$ the modulus of the complex permeability. This relation is valid only for low harmonic fields which is the case at $1x10^{-3}$ T induced field.

3. RESULTS AND DISCUSSION

3.1 Effect of the resin content

The effects of the resin content on the properties of iron-resin composites made with regular iron powders pressed at 620 MPa (45 tsi) and cured in air at 160°C for six hours are presented in this section. The effect of the resin content on density and electrical resistivity of iron-resin composite bars is presented in Figure 3. The density decreases as the resin content increases because the resin density is approximately seven times lower than that of iron. However, the relative densification ρ/ρ_{th}, where ρ_{th} is the theoretical density of the dielectromagnetic, increases as the resin content increases from 0.912 for the iron compact to 0.957 for the 1.8 wt% resin composites.

Figure 3 also shows that the electrical resistivity rapidly increases with an increase of the resin content indicating that the thickness of the resin layer between the iron particles increases. In fact, the resistivity of iron-resin composites containing 1.8 wt% resin is close to 2000 μΩ-m which is three orders of magnitude higher than that of iron compacts without resin (2 μΩ-m). However, it is important to note that the resistivity of iron compacts without resin is still high compared with bulk iron: 2 μΩ-m vs 0.1 μΩ-m respectively. This is likely due to the thin oxide layer present at the surface of the iron powder and the porosity in the compacted iron samples. The resistivity of iron compacts with no resin before curing, i.e., in the green state, is approximately 15 μΩ-m. This suggest that some electric contacts are created between the iron particles during the curing treatment at 160°C.

The effect of the resin content on the transverse rupture strength is presented in Figure 4. The strength of the composites increases with the resin content, reaching a maximum close to 125 MPa (18 x 10^3 psi) at about 1 wt% resin and then decreases. This suggests that the strength is mainly governed by the iron-resin bonding. An increase of the resin content beyond the minimum amount necessary to cover each iron particle does not further improve the strength. In this particular iron-resin system, the minimum quantity of resin required to cover the iron particles is approximately 1 wt%. It is believed that this quantity may depend on the surface area of the iron powder, i.e., shape and size of the iron particles.

The effect of the resin content on the apparent permeability measured at 60 and 400 Hz and at 1 Tesla of magnetization is presented in Figure 5. At both frequencies, the apparent permeability decreases as the resin content increases. The permeability drops from approximately 450 for the iron compact to 180 for the 1.8wt% resin composites. This can be attributed to the decrease in density or more specifically to the decrease of the filling factor (decrease of the volume occupied by iron). The length of the distributed air-gap (i.e., thickness of the resin layer between the iron powder) increases as the resin content increases leading to a shearing of the hysteresis curves. On the other hand, no significant differences are observed between the permeability measured at 60 Hz and 400 Hz.. This is likely due to the fact that the volume of iron penetrated by the electromagnetic wave does not change from 60 Hz to 400 Hz since the penetration depth of the electromagnetic wave is greater than the radius of the iron powder at these frequencies.

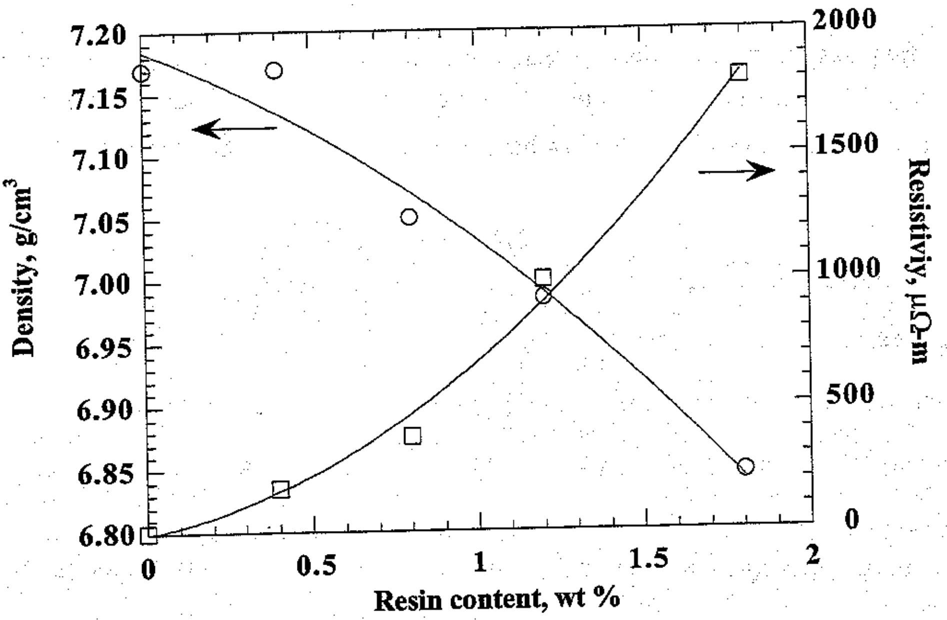


Figure 3: Effect of resin content on density and resistivity of iron-resin composites fabricated with regular powder pressed at 620 MPa (45 tsi) and cured at 160°C for 6h.

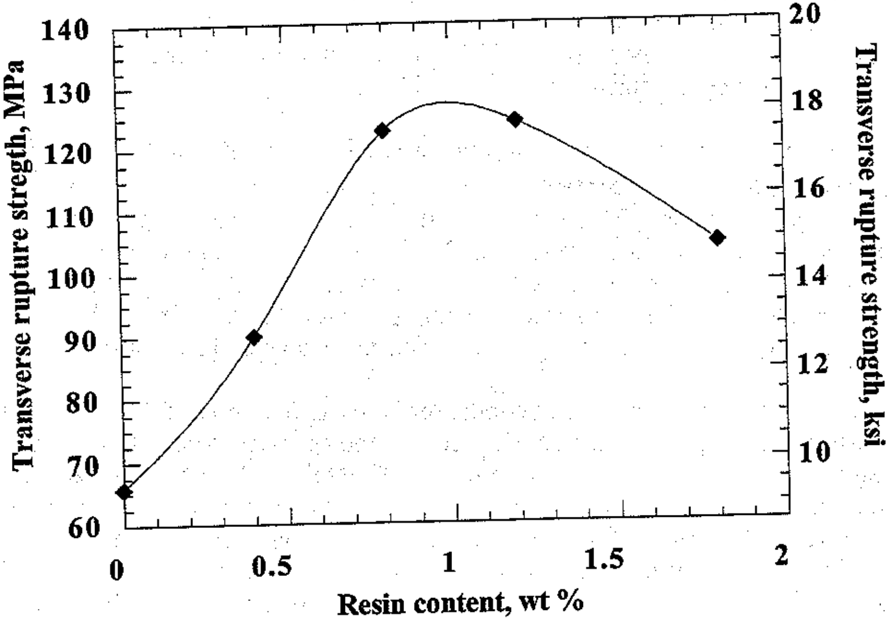


Figure 4: Effect of resin content on transverse rupture strength of iron-resin composites fabricated with regular powder pressed at 620 MPa (45 tsi) and cured at 160°Cfor 6h.

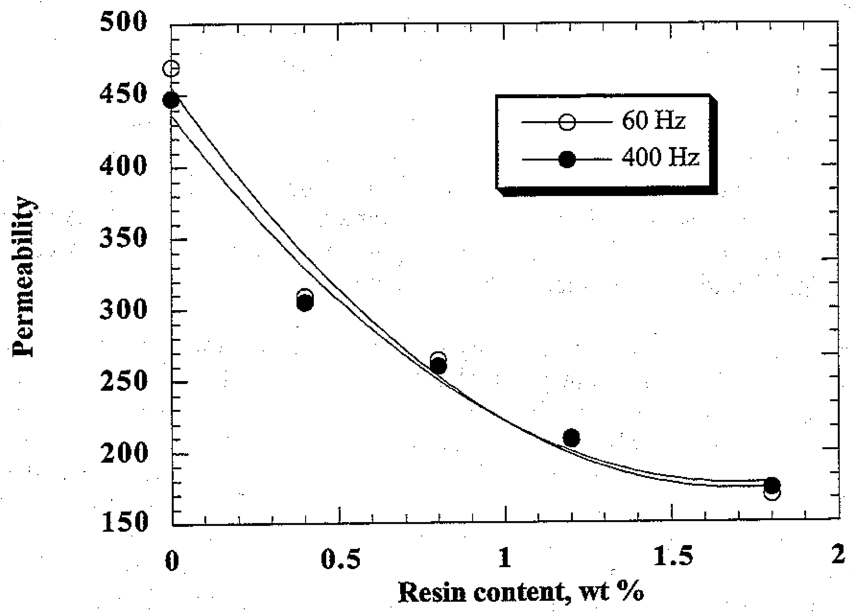


Figure 5: Effect of resin content on the apparent permeability at 60 Hz and 400 Hz and at 1 Tesla of magnetization of iron-resin composites pressed at 620 Mpa (45 tsi) and cured at 160°C for 6h.

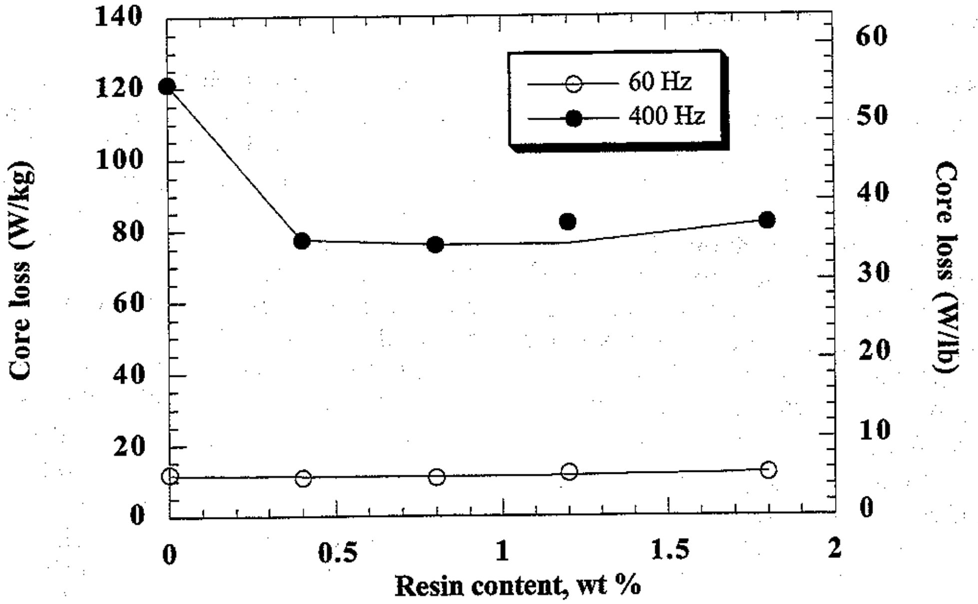


Figure 6: Effect of resin content on the apparent core losses at 60 Hz and 400 Hz and at 1 Tesla of magnetization of iron-resin composites pressed at 620 Mpa (45 tsi) and cured at 160°C for 6h.

The effect of the resin content on the apparent core losses measured at 60 and 400 Hz and at 1 Tesla of magnetization is presented in Figure 6. At 60 Hz, a total core losses of about 12 W/kg (5.4 W/lb) is measured for all the tested materials including the iron samples without resin. A.Kordecki et al. [16] have shown that at 1 Tesla and 50Hz, the hysteresis losses represent 95% of the total core losses in iron-0.5 wt% resin composite. It is then believed that the hysteresis losses do not significantly change when the resin content varies from 0wt% to 1.8wt%. Furthermore, the variation of the eddy current losses does not significantly affect the total core losses when the resin content varies from 0 to 1.8 wt%. At 60 Hz, the thin oxide layer present at the surface of the iron powder together with the small length of the eddy current path in the composite is sufficient to keep the eddy current losses low.

At 400 Hz, the total core losses of iron-resin composites are significantly lower than that of iron compacts without resin: approximately 80 W/kg (36 W/lb) and 120 W/kg (54 W/lb) respectively. At this frequency, the natural oxide layer on the particle surfaces is no longer sufficient to keep the losses low, thus a minimum amount of resin is needed. For this particular system, it seems that the insulation provided by 0.4 wt% resin (174 μΩ-m) is sufficient to keep the eddy current losses low and increasing the resin content will not significantly further reduce the total core losses at 400 Hz.

The effects of resin content on the initial permeability and specific losses measured at 1 mT from 1 kHz to 1 MHz are presented in Figures 7 and 8 respectively. Figure 7 shows that the initial permeability decreases as the resin content increases. This behavior is similar to the one described for the apparent permeability at low frequency and is related to the density or filling factor (volume occupied by iron) which decreases as the resin content increases (see Figure 3). For iron-resin composites, the initial permeability is constant up to a specific frequency after which it starts to drop. That critical frequency increases with an increase of the resin content. This can be explained by an improvement of the insulation between iron particles as the resin content increases which decreases the eddy currents while keeping permeability constant up to higher frequencies. For iron-resin composites containing 1.8 wt% resin, the eddy currents start to be important and affect the initial permeability at frequencies above 30 kHz. For the iron sample with no resin, eddy current effects are important at 1 kHz and the slope of the initial permeability curve is negative at that frequency. Specific losses are markedly reduced when a small amount of resin is added to the iron powder (Figure 8). The losses decrease with an increase of the resin content e.g., 1015μW/cc for iron-0.4 wt%resin to 428 μW/cc for iron-1.8 wt%resin at 100 kHz. This is in good agreement with equation 4 that states that an increase in electrical resistivity reduces the eddy current losses.

3.2 Effect of the iron powder particle size

Results obtained with iron-0.8% resin composites made with iron powders with different particle sizes (regular, coarse and fine) pressed at 620 MPa (45 tsi) and cured in air at 160°C for six hours are presented in this section. The effect of the iron particle size on the density, electrical resistivity and strength is presented in Figure 9. The density of the iron-0.8% resin composites increases slightly as the average iron particle size increases (Fig. 9a). This can be partly due to the fact that inter-particle friction and bridging effects are usually more important during the compaction of fine particles [17]. Furthermore, work hardening that occurs during compaction is probably less pronounced for coarse particles since the dislocation slip distances are proportional to the powder size [18]. The electrical resistivity increases as the average iron particle size decreases: resistivity is approximately 260 μΩ-m for coarse particles and 460 μΩ-m for fine particles. This is attributed to the number of insulated inter-particle contacts which increases as the powder size decreases. The transverse rupture strength also varies with particle size (Fig. 9b). A strength of 82 MPa (12 x 10^3 psi) is obtained for composites made with fine iron powders while approximately 125 MPa (18 x 10^3 psi) is achieved for composites made with regular or coarse iron powders. This difference can be related to the density and the amount of resin per surface area. Fine iron powder composites have a lower density compared to the regular and coarse powder composites and lower density usually leads to lower strength. For a given quantity of resin, the amount of resin per surface area is lower when the surface area of the powder increases. The total surface area is larger for the fine iron powder compared with the regular or coarse iron powder. The amount of resin used (0.8wt%) is probably not high enough to completely cover the surface of the fine iron powder. The amount of resin

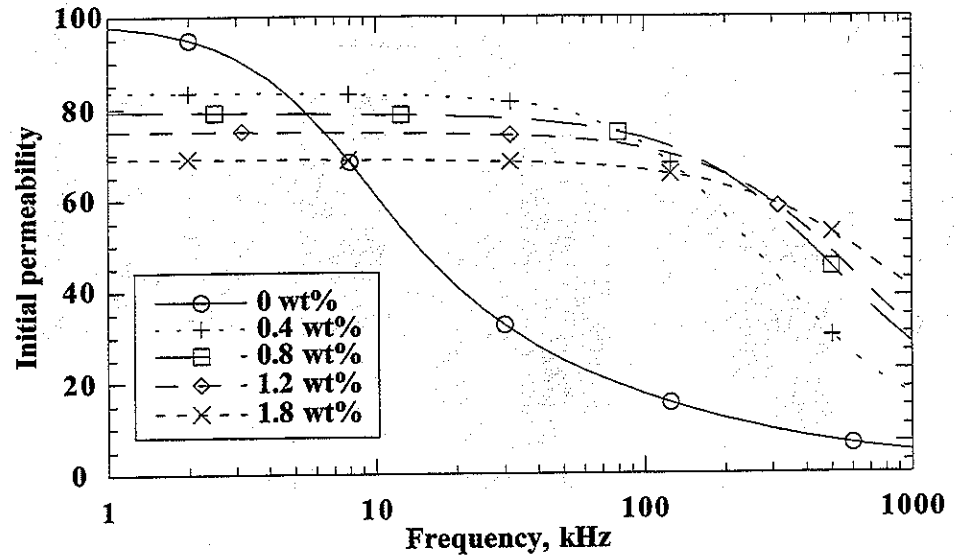


Figure 7: Effect of the resin content on the initial permeability from 1 kHz to 1 Mhz for iron-resin composites pressed at 620 MPa (45 tsi) and cured at 160°C for 6h.

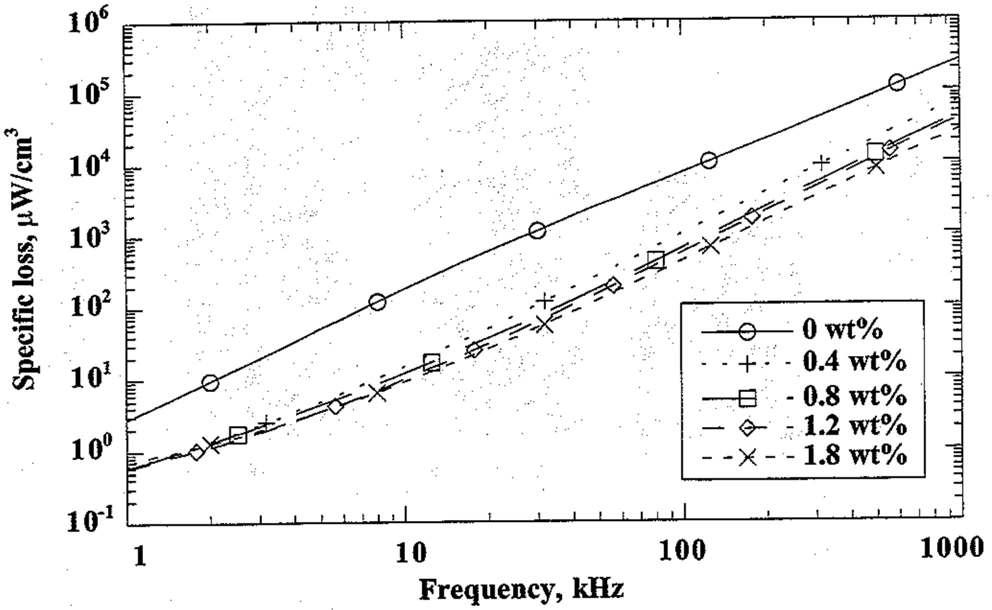
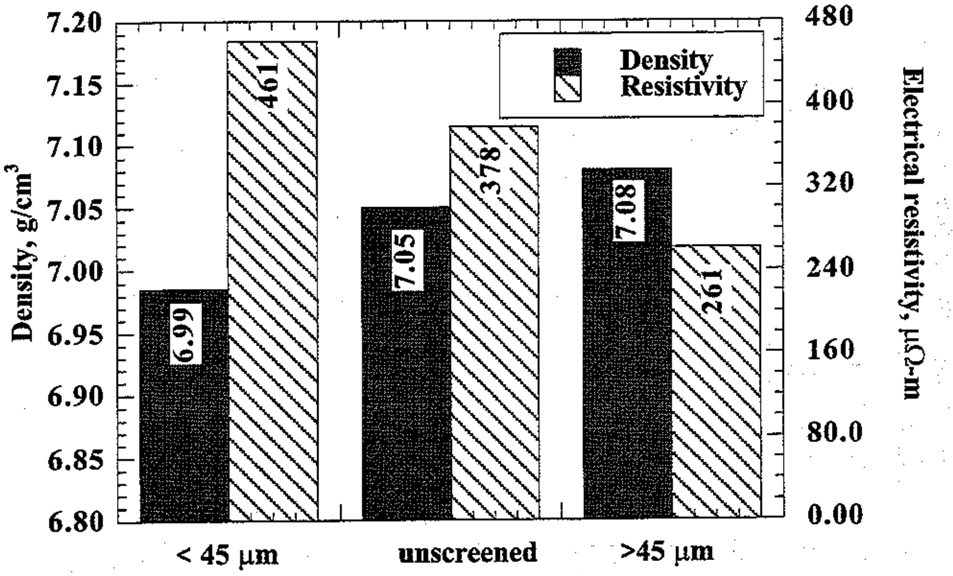
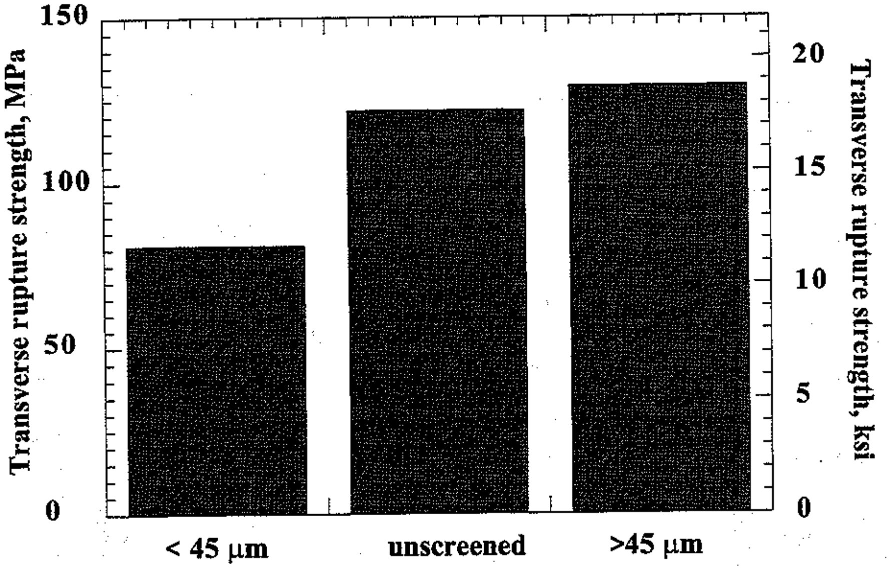


Figure 8: Effect of the resin content on the specific core losses at 1 mT from 1 kHz to 1 Mhz for iron-resin composites pressed at 620 MPa (45 tsi) and cured at 160°C for 6h.



a)



b)

Figure 9: Effect of the iron powder particle size on properties of iron-0.8wt%resin composites pressed at 620 Mpa (45 tsi) and cured in air at 160°C for 6 hours: a) density and electrical resistivity b) transverse rupture strength.

necessary to optimize the strength of the composites must then be adjused to the specific surface area of a given powder. Green strength from mechanical interlocking is also enhanced by an irregular particle shape [18]. Since the fine iron particles contained more spherical particles, it is possible that mechanical interlocking is lower for these particles.

The effect of the iron particle size on the apparent permeability measured at 60 and 400 Hz and at 1 Tesla of magnetization is presented in Figure 10. The permeability does not significantly change from 60 Hz to 400 Hz. The apparent permeability is lower for composites fabricated with fine powders (175) compared to composites made with regular or coarse powders (265). This is partly due to differences in density and in magnetic domain sizes. The density is lower and the domain sizes are smaller for fine powder composites as opposed to coarse and regular powder composites.

The effect of iron particle size on the apparent core losses measured at 60 and 400 Hz and at 1 Tesla of magnetization is presented in Figure 11. At 60 Hz and 400 Hz, core losses for iron-0.8% resin composites made with fine iron powders are higher than those made with regular or coarse iron powders even if the resistivity of the former is higher. Beyond a critical threshold, the resistivity is not of prime importance at these low frequencies. As discussed in the previous section, the resistivity of iron-0.8 wt% resin composites is sufficient to keep the eddy current losses low. The differences observed in the total core losses in Figure 11 are therefore likely due to the hysteresis losses. Several authors [19,20] confirm a linear relationship between coercive force H_c and the grain size d_g for otherwise constant conditions:

$$H_c \propto \frac{k}{d_g} \tag{6}$$

For water-atomized iron powders, the grain size is inversely proportional to the cooling rate which decreases with an increase of the particle size. Hysteresis losses are then higher for fine powder particles due to their smaller grain sizes. Hysteresis losses also depend on the stress state. Internal stresses are probably higher in composites fabricated with fine iron powders. During compaction, work hardening is more important in composites made with fine powders because the dislocation slip distances are shorter in fine powders [18]. Internal stress measurements should therefore be done to confirm this hypothesis.

Figure 12 and 13 show the initial permeability and specific losses at high frequency (1 kHz to 1 MHz) and 1 mT of magnetic induction for iron-0.8 wt% resin composites fabricated with fine, regular and coarse iron powders. Fine iron powder composites have lower initial permeability than regular and coarse iron powder composites: approximately 70 and 80 respectively. This is partly related to their density (see figure 9). In fact, the lower the density, the lower the initial permeability. On the other hand, Figure 12 shows that the initial permeability of the fine iron powder composite is more stable versus frequency than that of the regular and coarse iron powder composites. There is no significant difference between the initial permeability of regular and coarse iron powder composites. Initial permeability decreases due to the rise of eddy currents. Eddy currents decrease as the resistivity increases and the length of the eddy current path decreases. The length of the eddy current path is shorter and the resistivity is higher for fine powders (see Figure 9) hence the permeability remains stable up to higher frequencies. According to Figure 12, eddy current effects begin to be significant at approximately 30 kHz and 100 kHz for respectively the regular or coarse powder and the fine iron powder composites.

Specific losses are lower in fine iron powder composites than for regular or coarse iron powder composites (Figure 13). At 1 kHz, the differences in core losses are small but they increase at higher frequency. Losses in the coarse and regular iron powder composites are very similar at high frequencies. This is probably due to the fact that the most important contribution to the eddy current losses is due to the coarse powder and that the regular powder is composed of 73wt% coarse powder.

For the experimental conditions used in this study, the iron particle size had a more important effect than the resin content on the magnetic properties at high frequency. For composites with similar initial permeability (~ 70), specific losses at 100 kHz in iron-0.8 wt% resin composites made with fine iron

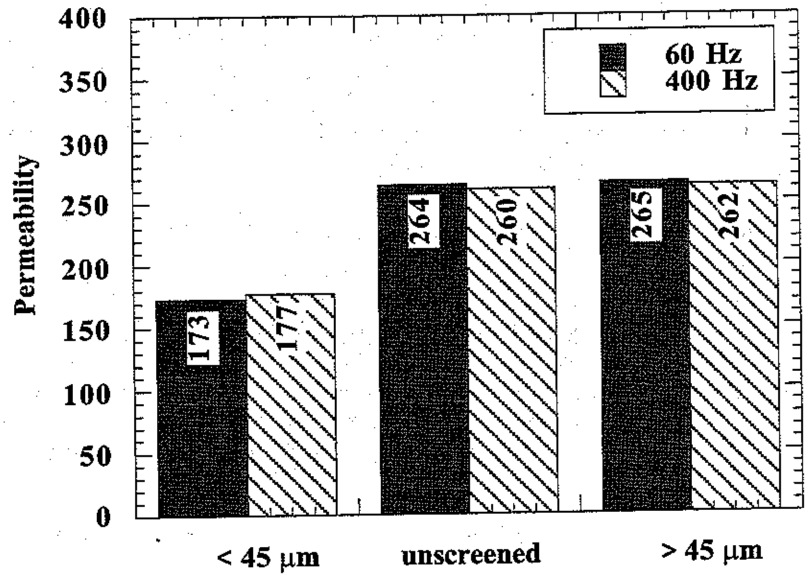


Figure 10: Effect of the iron particle size on the apparent permeability at 60 Hz and 400 Hz and at 1 Tesla of magnetization of iron-0.8 wt% resin composite pressed at 620 MPa (45 tsi) and cured at 160°C for 6h.

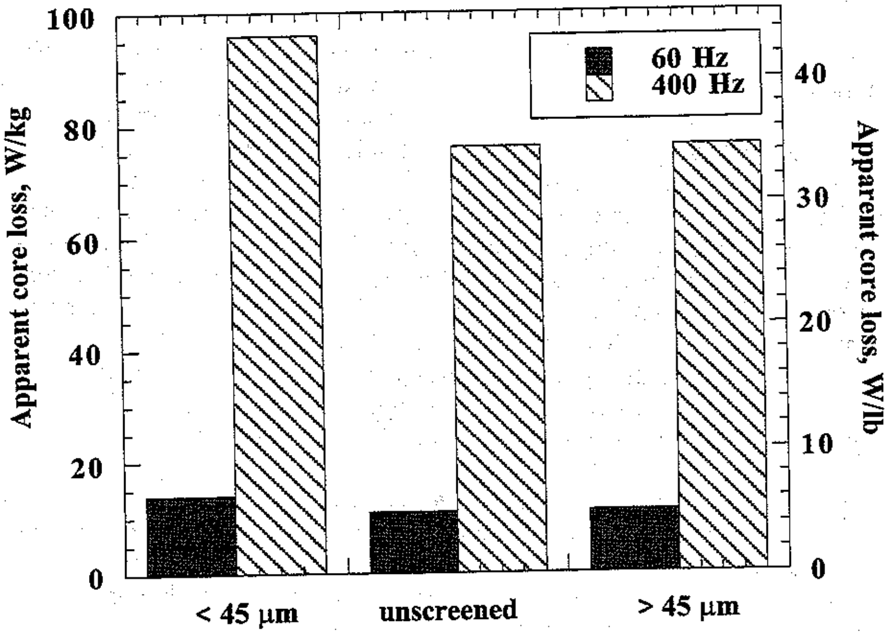


Figure 11: Effect of the iron particle size on the apparent core loss at 60 Hz and 400 Hz and at 1 Tesla of magnetization of iron-0.8 wt% resin composite pressed at 620 MPa (45 tsi) and cured at 160°C/6h.

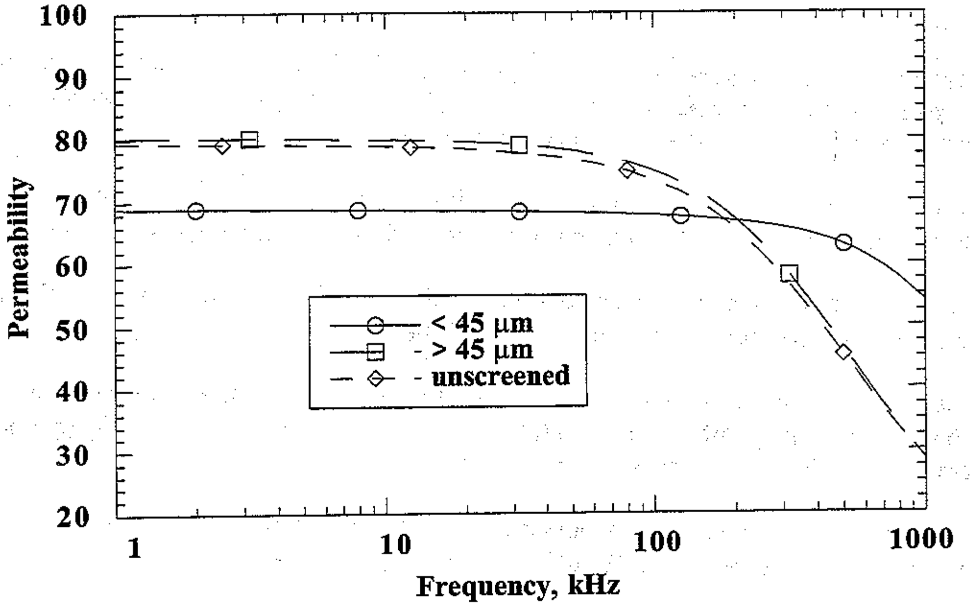


Figure 12: Effect of the iron particle size on the initial permeability from 1 kHz to 1 MHz for iron-0.8 wt% resin composites pressed at 620 MPa (45 tsi) and cured at 160°C for 6h.

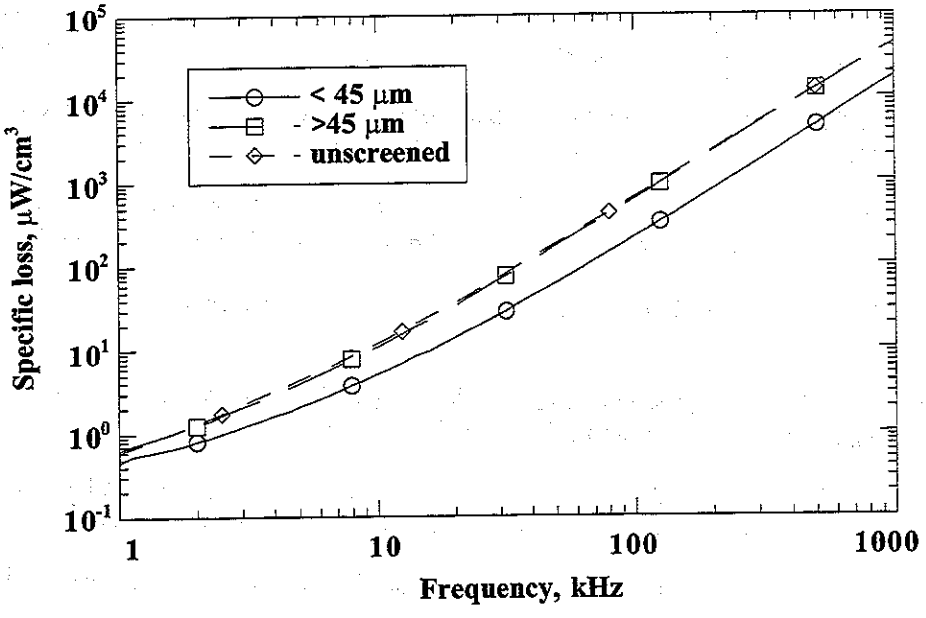


Figure 13: Effect of the iron particle size on the specific core losses from 1 kHz to 1 MHz and at 1 mT for iron-0.8 wt% resin composites pressed at 620 MPa (45 tsi) and cured at 160°C for 6h.

powders are lower than in iron-1.8 wt% resin composites made with regular iron powders: 211 and 428 μW/cm^3 respectively (see Fig. 8 and 13).

CONCLUSIONS

The effects of iron particle size and resin content on physical, mechanical and magnetic properties of iron-thermoset resin composites pressed at 620 MPa (45 tsi) and cured in air at 160°C for 6 hours were evaluated. The results lead to the following observations and conclusions:

- The transverse rupture strength depends on the iron particle size and the resin content. For a given iron particle size distribution, the strength can be maximized by adjusting the resin content.
- The resin content should be kept as low as possible to achieve high permeabilities. Use of a fine iron powder decreased both the apparent and initial permeability.
- At 60 Hz, the resin content did not have a significant effect on core losses. At 400 Hz, eddy currents are greater and a minimum quantity of resin had to be used to keep the losses low. Using a fine iron powder increased core losses at these low frequencies.
- At high frequencies, eddy currents and the losses decreased as the resin content increased. Using fine iron powder markedly decreased the losses at high frequency.

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