IRON-RESIN COMPOSITES FOR HIGH FREQUENCY AC MAGNETIC APPLICATIONS.

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ABSTRACT

Dielectromagnetics such as iron-resin materials are composites designed for AC magnetic applications. The composition and processing of these iron-resin powders can be adjusted to meet specific application requirements. It has previously been demonstrated that the dry mixing of a unique thermoset resin with a broad particle size distribution of a pure iron powder permitted to produce dielectromagnetics with very good magnetic performances at low frequencies: high permeability and low core loss. However, it is known that as the frequency increases, a more efficient insulation between the iron particles, combined with a finer particle size distribution of that iron powder is necessary to minimize eddy currents.

Two mixing techniques were tested for manufacturing iron-resin powders for high frequency magnetic applications. The particle size distribution of the base iron powder and the resin content were also varied. This paper presents the physical, mechanical and magnetic characterizations of specimens produced from these materials.

INTRODUCTION

Conventional P/M is a well recognized route for the manufacturing of soft magnetic parts via the sintering process for DC magnetic applications. Recent improvements in materials (iron powders and polymers) and processes allowed the manufacturing of new dielectromagnetics for AC magnetic applications. For example, ATOMET EM-1, an iron-resin composite powder has been developed for AC applications in the low to medium frequency range (up to about 50 kHz). This iron-resin powder, conventionally pressed and cured at 175°C in air for one hour, produces parts with good AC magnetic properties and structural integrity [1]. These parts exhibit isotropic thermal and magnetic properties which allow for new designs and applications, especially in the automotive industry [2]. However, high frequency AC magnetic applications require different materials and properties.

For instance, core loss and initial permeability are the most important property requirements that have to be considered when selecting materials for high frequency applications. It is well-known that as the frequency increases, the relative importance of eddy current losses on total core loss increases and most of the improvement in core loss at high frequencies comes from improvements in eddy current shielding. The relationship between material characteristics and eddy current losses (P_e) is given in the following equation:

$$P_e = \frac{K_e B^2 f^2 d}{\rho} \qquad (1)$$

where K_e is a constant, B is the magnetic induction, f is the frequency, d is the shortest dimension perpendicular to the flux path and ρ is the electrical resistivity. The last two parameters are materials characteristics that can be modified. In the case of laminations, this can be achieved by alloying iron with silicon (higher resistivity), by using thinner laminations and/or adding an insulating layer between laminations. In the case of dielectromagnetics, d and ρ can be modified by varying the degree of insulation between the iron particles and by varying their particle size.

In order to better understand the relationship between the material processing and its properties, a detailed study was undertaken. In this paper, the effects of the powder mixing process, particle size distribution of the base iron powder and resin content on both the mechanical and AC magnetic properties are evaluated and discussed.

EXPERIMENTAL PROCEDURE

Iron-resin mixes were made from a high purity water-atomized iron powder (ATOMET 1001HP) that was screened in two different particle size fractions. The screen analyses are given in Table I. A first fraction containing 96 wt% of particles passing through a 200 mesh US sieve is identified "<75 μm" and a second fraction containing 89 wt% of particles passing through a 325 mesh US sieve is identified "<45 μm". Iron-resin mixes were prepared according to two methods: a conventional mixing technique in which the phenolic resin powder was admixed to the iron powder and a wet blending technique. The latter process consisted of dissolving the resin in an adequate quantity of solvent, pouring the solution into a beaker containing the iron particles, mixing and then drying. Three levels of resin content were evaluated in the dry and wet mixes made from the <75 μm and <45 μm iron powders: 0.8, 1.0 and 1.2 wt% phenolic resin. Thus in this study, twelve mixes of iron-resin composites were evaluated for their physical, mechanical and magnetic properties.

Table I. Particle size distribution of the two types of iron powders tested in this study.

Screen analysis, wt%		Powder identification	
U.S. Sieve	Microns	<75 μm	<45 μm
+140 Mesh	+105 μm	Trace	
-140 +200	-105 +75	3.9	
-200 +230	-75 +63	14.2	Trace
-230 +270	-63 +53	11.0	0.8
-270 +325	-53 +45	26.2	10.0
-325 +400	-45 +38	11.2	14.6
-400 Mesh	-38 μm	33.5	74.6

For each of the mixes, five rectangular transverse rupture strength bars (TRS bars) and one ring (5.26 cm OD, 4.34 cm ID and 0.635 cm thick) were pressed at 620 MPa (45 tsi) and 65°C and cured at 175°C for one hour in air to fully cross-link the resin and increase the mechanical strength of the composite specimens. In order to achieve the highest possible density and strength, there was no admixed lubricant and the die walls were lubricated using a graphite spray. The density (absolute and relative), TRS, and electrical resistivity were measured on the TRS bars while the AC magnetic properties at high frequencies were measured on rings. Transverse rupture strength tests were made according to MPIF standard 41 and 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. Five readings were taken on the top and bottom faces of each TRS bar and averaged.

An AC magnetic characterization at high frequency was carried out on rings wound with 96 turns of a 24 gauge insulated copper wire. The initial permeability was evaluated from 1 kHz to 1 MHz using a HP4192A LF impedance analyzer equipped with a HP16047A test fixture. An excitation of 1 V_{RMS} producing a field $B < 5x\ 10^{-4}$ T (5 Gauss) was applied. The specific losses $P(\omega)$ at 1 mT (10 Gauss) were also evaluated using the following relation:

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

where ω is the frequency ($2\pi f$), μ_0 is the free space permeability ($4\pi \times 10^{-7}$ H/m), μ'' is the imaginary part of the permeability, $|B^*|$ is the modulus of the induced magnetic field and $|\mu^*|$ the modulus of the complex permeability. This relation stands for low harmonic fields which is the case for an induced field of 1 mT.

RESULTS AND DISCUSSION

Physical and mechanical properties

The effects of the resin content, iron particle size distribution (<75 μm and <45 μm) and powder processing technique (dry and wet mixing) on the density of iron-resin composite bars pressed at 65°C/45 tsi (620 MPa) and cured in air at 175°C for one hour are presented in Figure 1. As expected, an increase of the resin content decreases the density of the pressed bars (Fig. 1a) because of the low specific gravity of the resin. For instance, when the resin content increases from 0.8% to 1.2% there is a density drop of approximately 0.10 g/cm^3 for bars made from wet mixed powders and 0.05 g/cm^3 for those made from dry mixed powders. However, the relative density of the composite bars, defined as the cured-to-theoretical density ratio (Fig. 1b), increases with an increase in the resin content from 0.8% to 1.2%: an increase of approximately 0.3% for bars made from wet mixed powders and 1.25% for those made from dry mixed powders. This large difference is related to the fact that for a given amount of resin in the powders, the wet mixing process produced a slightly higher density than the conventional dry mixing process. This is especially true at 0.8% resin and the effect diminishes with an increase of the resin content. Accordingly, the relative density of bars made from wet mixed powders is higher and more constant with respect to the resin content than that of bars pressed from dry mixed powders.

Also, as-expected, the <75 μm powder is more compressible than the <45 μm powder. Indeed, for any resin content and powder processing, bars pressed from the former powder gave densities 0.05 g/cm^3 higher than bars pressed from the latter. This is partly due to the fact that a fine particle size creates a higher friction during compaction thus making pressing to a high density more difficult [3].

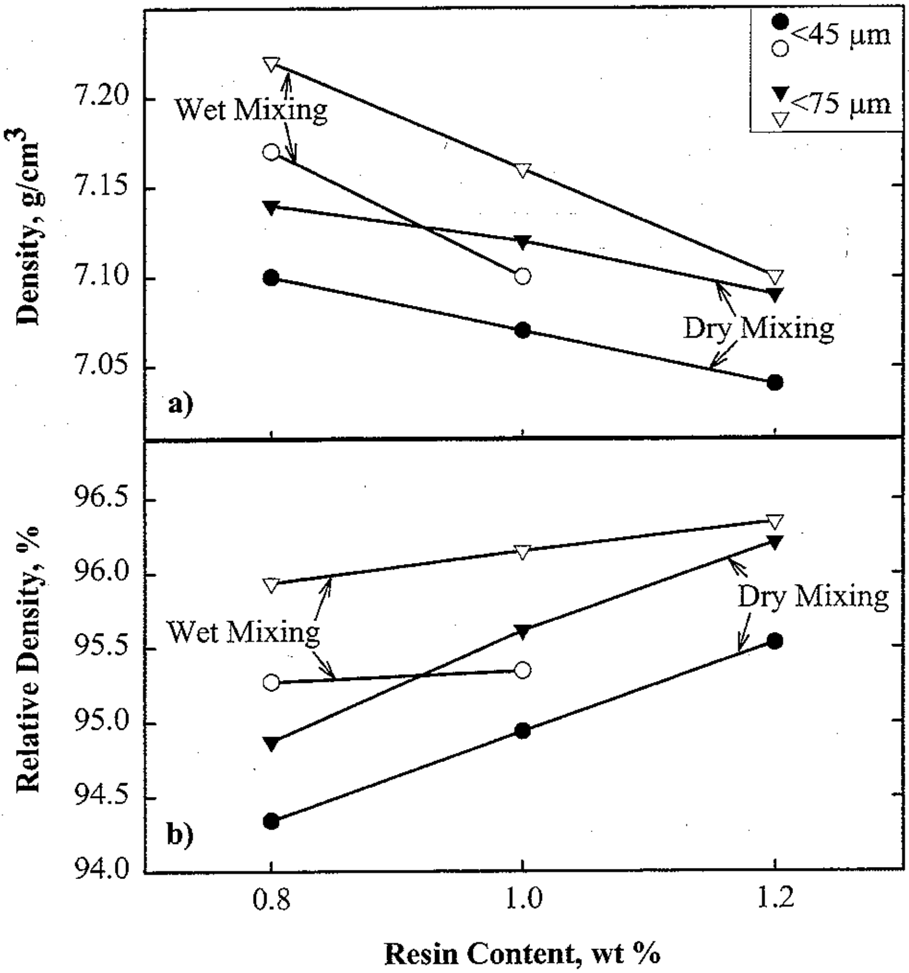

Figure 1. Effects of the resin content, iron particle size distribution and mixing technique on density (a) and relative density (b) of iron-resin composites pressed at 65°C/45 tsi (620 MPa) and cured in air at 175°C/1 h.

The effects of the resin content, iron particle size distribution (<75 μm and <45 μm) and powder processing technique (dry and wet mixing) on the TRS of iron-resin composite bars pressed at 65°C/45 tsi (620 MPa) and cured in air at 175°C for one hour are presented in Figure 2. A broad range of strengths was obtained with these composites: from 13000 psi up to 21000 psi (90 to 145 MPa). The effect of the resin content and particle size depends on the mixing process. Generally, the dry mixed powder gave composites with a higher strength than those made from a wet mixed powder. For instance, for the dry mixing process, a maximum of around 21000 psi or 145 MPa in strength is achieved by using 1.0% to 1.2% resin in the composites whatever the iron particle size used. In the case of the wet mixing process, a more complicated trend is observed. For the <75μm iron powder particle size, the strength continuously increases with the amount of resin while for the <45μm iron particle size, the highest value occurs at around 0.8% resin in the composites. This behavior may be related to the size and distribution of the porosity in the pressed composites and in the way the resin coats the iron particles and fills this porosity. It seems that for a given combination of powder processing and iron powder particle size, there is an optimum amount of resin to maximize the strength. A similar behavior had already been observed in the case of dielectomagnetics with different iron powder particle sizes [4].

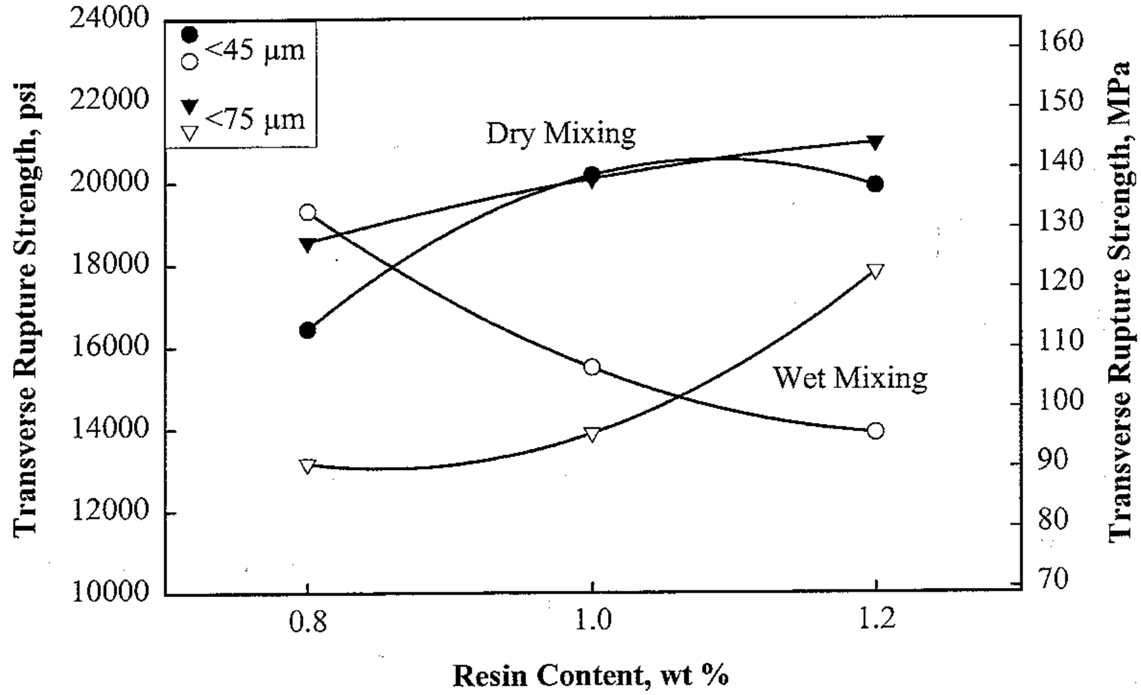

Figure 2. Effect of the resin content, iron particle size distribution and mixing technique on the transverse rupture strength of iron-resin composites pressed at 65°C/45 tsi (620 MPa) and cured in air at 175°C/1 h.

The effects of the resin content, iron particle size and powder processing technique on the electrical resistivity of iron-resin composite bars pressed at 65°C/45 tsi (620 MPa) and cured in air at 175°C for one hour are presented in Figure 3. As expected, the resistivity of the composites increases with the resin content: it almost doubles by increasing the resin content from 0.8% to 1.2%. The iron particle size does not have a significant impact on the resistivity of bars made from dry mixed powders but for bars made from wet mixed powders, the <45 μm powder gives composites with a two to four-fold increase in resistivity compared with the <75 μm powder. The resistivity of the composites processed by dry mixing is relatively high but, as expected, it is much higher when they are processed by wet mixing. For example, the resistivity of bars made from wet mixed composite powders varies from 0.62 to 3.2 mohm-m compared to 0.14 to 0.26 mohm-m for bars made from dry mixed powders. It corresponds to an increase in resistivity of approximately one order of magnitude.

It has already been reported that similar composites processed by dry mixing yielded a relatively high resistivity, in the 0.15 to 0.20 mohm-m, in both the as-pressed and after curing states [1]. This is possible because the specific thermoset resin used in these experiments has a very good capability to be intimately mixed with the iron particles during the dry mixing process and also to wet the iron particles during pressing and curing steps which follow. The range of resistivity achieved with the dry processed composites is adequate for applications at low to medium frequency. However, for applications at higher frequencies, higher resistivities are required in order to minimize eddy currents and this is exactly what the wet mixing process provides: an improvement of the insulation. The wet process improves the uniformity of the resin coating and consequently it increases the thickness of the insulating layer between adjacent iron particles. This improved insulation now benefits from the use of a finer particle size distribution of the iron, which is not observed with dry mixed composite powders. For wet mixed powders, it appears that the finer the particle size of the resin-coated iron, the greater the number of insulating contacts for a given electrical path and, consequently, the higher the overall resistivity.

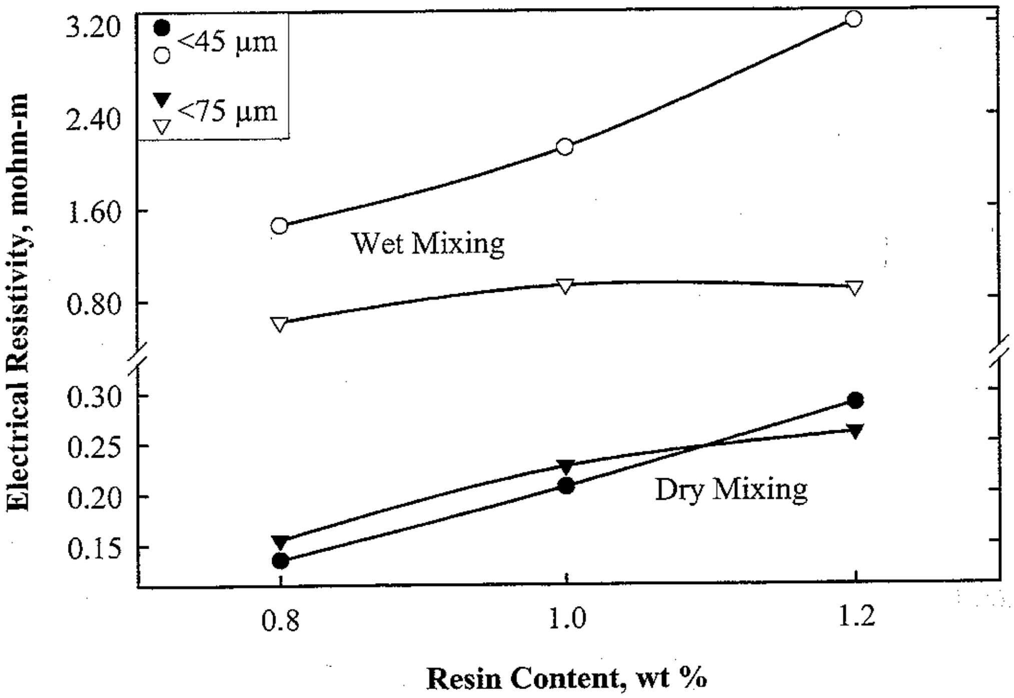

Figure 3. Effects of the resin content, iron particle size distribution and mixing technique on the electrical resistivity of iron-resin composites pressed at 65°C/45 tsi (620 MPa) and cured in air at 175°C/1 h.

AC magnetic properties

The effects of the frequency, iron particle size distribution, amount of resin and mixing technique on the initial permeability (1 kHz to 1 MHz) of iron/resin composite rings pressed at 65°C/45 tsi (620 MPa) and cured in air at 175°C for one hour are presented in Figure 4. For composites containing 0.8% resin (Fig. 4a), results show that the initial permeability decreases when the particle size decreases especially for those pressed from dry mixed powders. This behavior is related to the density which also decreases when the particle size decreases. The mixing technique affects the initial permeability which drops from 76-78 for dry processed composites down to 71-72 for wet processed composites. Because the resin is more uniformly distributed in the wet mixed composites, the thickness of the insulating layer between iron particles increases. This is equivalent to an increase in the effective distributed air gaps between iron particles which decreases the overall permeability.

The stability of the initial permability with respect to frequency increases with the degree of insulation between the iron particles and a decrease in the particle size. This behavior is directly related to the degree of efficiency in which iron particles are shielded against eddy currents. Eddy currents induce an opposing magnetic field in the samples that reduces their apparent permeability. Accordingly, the iron/0.8% resin composite that had the most stable permeability was made from <45 μm iron powder processed by wet mixing.

The effect of the resin content on the initial permeability is shown in Figure 4b for the composites made from <45μm iron powder processed by wet mixing. In this case, an increase of the resin content decreases the initial permeability from 71 to 56 for composites containing 0.8% and 1.2% resin respectively. This is related to a decrease in the density and an increase in the effective distributed air gaps.

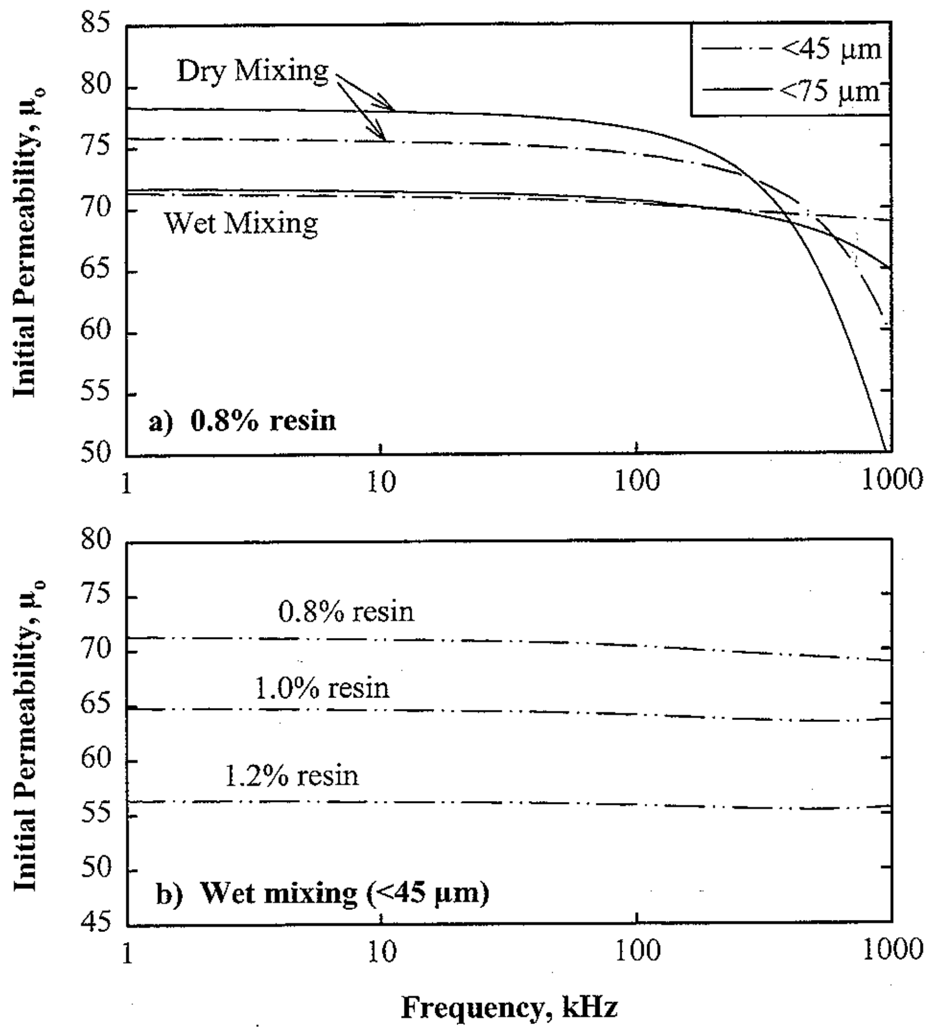

Figure 4. Effect of frequency on the initial permeability of iron/resin rings pressed at 65°C/45 tsi (620 MPa) and cured in air at 175°C/1 h: a) effects of iron particle size and mixing technique for composites containing 0.8% resin; b) effect of the resin content for composites made from <45 μm iron powder processed by wet mixing.

The effects of the frequency, iron particle size distribution, amount of resin and mixing technique on the total losses calculated at 10 G (1 mT) of iron/resin composite rings pressed at 65°C/45 tsi (620 MPa) and cured in air at 175°C for one hour are presented in Figure 5. For composites containing 0.8% resin (Fig. 5a), results show that at low frequency, all the composites perform about the same but as the frequency increases differences in losses between composites appear. At a given frequency and for a given amount of resin in the composites, losses decrease by improving the degree of insulation between the iron particles through wet processing and by decreasing the particle size of the iron powder. This behavior is again directly related to the degree of efficiency in which iron particles are shielded against eddy currents (see equation 1). Accordingly, for a given magnetic induction and frequency, the lowest eddy current losses would be achieved for composites made from <45 μm iron powder (smallest particle size) processed by wet mixing (highest resistivities). For example, the lowest and highest total core loss values (at 10 G and 100 kHz) reported in Figure 5a for iron/0.8% resin rings are 284 $\mu W/cm^3$ and 101 $\mu W/cm^3$ for composites made from <75 μm iron powder processed by dry mixing and <45 μm iron powder processed by wet mixing respectively.

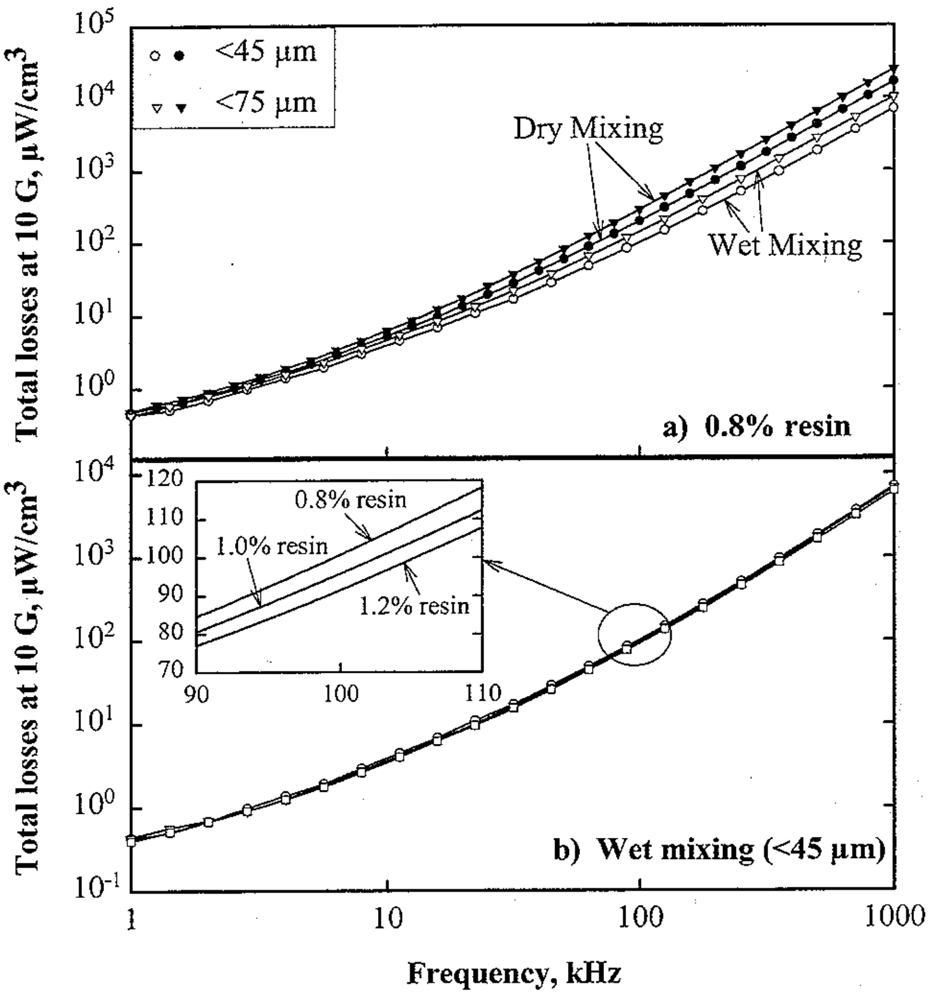

Figure 5. Effect of frequency on total losses at 10 G of iron/resin rings pressed at 65°C/45 tsi (620 MPa) and cured in air at 175°C/1 h: a) effects of iron particle size and mixing technique for composites containing 0.8% resin; b) effect of resin content for composites made from <45 μm iron powder processed by wet mixing (the inset shows an enlargement in the 100 kHz frequency range).

The effect of the resin content on the total losses at 10 G is shown in Figure 5b for composites made from <45μm iron powder processed by wet mixing. On this logarithmic scale, the products are very similar. However, an enlargement of that figure in the 100 kHz frequency range (inset of Fig. 5b) shows that an increase of the resin content slightly decreases the total loss at 10 G. For example, at 100 kHz, total losses are 101 $\mu W/cm^3$ and 92 $\mu W/cm^3$ in composites containing 0.8% and 1.2% resin respectively. In fact, the 9% improvement in core loss achieved by increasing the resin content corresponds to a drop in initial permeability of 21% (see Fig. 4b).

CONCLUSION

In this study, the effects of the powder mixing technique, particle size distribution of the base iron powder and resin content on the mechanical and AC magnetic properties of iron-resin composites were evaluated. For high frequency applications, the following key parameters have been identified:

1. A wet mixing technique is preferred to dry mixing. Iron-resin composites prepared by wet mixing have a much higher resistivity, a lower but more stable initial permeability as a function of frequency and a lower core loss.

2. A fine particle size distribution of the iron powder is also preferred because it produces iron-resin composites with a higher resistivity together with a more stable permeability as a function of frequency and a lower core loss.

3. Compared with the effect of the iron particle size, an increase of the resin content between 0.8% to 1.2% has a smaller impact on the properties. Increasing the resin content decreases the density, slightly increases the resistivity, decreases the initial permeability by approximately 20% and improves the core loss by 9%.

Finally, this work showed the possibility of manufacturing iron-resin composite materials for high frequency magnetic applications. For instance, by combining a fine iron powder (<45 μm) with 0.8% phenolic resin using a wet mixing technique, iron-resin composites with the following properties were obtained after pressing at 65°C/45 tsi (620 MPa) and curing in air at 175°C/1 h:

- Electrical resistivity around 1.5 mohm-m.
- Initial permeability around 70.
- Permeability stable up to 1 MHz.
- Core loss around 100 $\mu W/cm^3$ at 100 kHz and 10 G.
- Mechanical strength in the 18000 to 20000 psi range (124 to 138 MPa).

REFERENCES

1. C. Gélinas, F. Chagnon and S. Pelletier, "Development of an Iron-Resin Composite Material for Soft Magnetic Applications", *Advances in Powder Metallurgy & Particulate Materials - 1996*, Compiled by Terry M. Caddle and K.S. Narasimhan, Metal Powder Industries Federation, Princeton, NJ, 1996, pp 20-85 to 20-97.

2. C. Gélinas, L.-P. Lefebvre, S. Pelletier and P. Viarouge, "Effect of Temperature on Properties of Iron-Resin Composites for Automotive Applications", *SAE Technical Paper Series 970421*, SAE International Congress & Exposition, Detroit, February 24-27, 1997.

3. R.M. German, *Powder Metallurgy Science*, Metal Powder Industries Federation, Princeton, NJ, 1984, p. 131.

4. L.-P. Lefebvre, S. Pelletier, B. Champagne and C. Gélinas, "Effect of Resin Content and Iron Powder Particle Size on Properties of Dielectromagnetics", *Advances in Powder Metallurgy & Particulate Materials - 1996*, Compiled by Terry M. Caddle and K.S. Narasimhan, Metal Powder Industries Federation, Princeton, New Jersey, 1996, pp 20-47 to 20-61.