EFFECT OF CURING CONDITIONS ON PROPERTIES OF IRON-RESIN MATERIALS FOR LOW FREQUENCY AC MAGNETIC APPLICATIONS.

C. Gélinas, F. Chagnon, S. Pelletier* and L.-P. Lefebvre*.

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

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

ABSTRACT

Iron-resin materials produced by dry mixing a unique thermoset resin with pure iron powders are good dielectromagnetics for pulsed DC and AC magnetic applications. These materials are cured at low temperature in order to melt the resin and make it flow around the iron particles increasing the strength of the composites. The electrical resistivity resulting from that resin insulation minimizes eddy current losses. Depending on the temperature of the curing treatment, stress relief may also contribute to further reduce the hysteresis loss.

A study was conducted to evaluate the robustness of the iron-resin system with respect to the curing conditions and their potential internal stress relieving effect. The highest mechanical strength is obtained by curing in the 200°C to 250°C range which gives composites with a high electrical resistivity and good magnetic properties in the 60 to 400 Hz range and beyond. For applications at 60 Hz, curing the composites at a higher temperature effectively minimizes the hysteresis loss. By curing at 400°C for 30 minutes in air the magnetic properties are optimized with a decrease of 12% in total core loss and an increase of 25% in AC permeability.

INTRODUCTION

The iron-resin composite powder ATOMET EM-1* has been developed for pulsed DC and AC magnetic applications at frequencies up to about 50 kHz. By pressing the iron-resin powder and curing at 175°C in air for one hour, parts with good AC magnetic properties and structural integrity are produced [1]. The curing allows the resin to melt and flow around the iron particles thus forming a composite structure having high electrical resistivity and mechanical strength.

For applications at 60 Hz, the degree of insulation required is minimal in this type of materials since most of the core loss does not originate from eddy currents but rather from the hysteresis loss [2]. This latter is

* Manufactured by Quebec Metal Powders Limited.

affected by the chemistry, the size of the magnetic domains and the internal stresses induced in the particles during compaction [3]. It could be possible to relieve these stresses by heat treating in the recrystallization temperature range which is typically between 570°C and 775°C for pure iron [4]. In the case of silicon steel laminations, it is common to make a stress relief treatment at about 800°C for one hour to improve the AC magnetic properties [5].

This study has been conducted to evaluate the effects of curing conditions on mechanical and magnetic properties. Curing temperatures as high as 500°C were reached in order to see if it is possible to stress relieve the compacts while maintaining an acceptable strength and resistivity for applications at low frequency. This paper presents the physical, mechanical and magnetic properties of iron/0.8% resin specimens, pressed at 65°C/45 tsi (620 MPa) and cured in air at different temperatures and periods of time.

EXPERIMENTAL PROCEDURE

A coarse high purity water-atomized iron powder was used in these experiments. Mixes were prepared by dry blending the coarse iron powder admixed with 0.8 wt% phenolic resin powder. The chemical and physical characteristics of the base iron powder are given in Table I. When admixed with the resin, the powder had an apparent density of 2.70 g/cm³ and a flow rate of 31 s/50 g.

Table I. Chemical and physical characteristics of the base iron powder used in this study.

Chemical analysis, wt%		
Carbon		0.004
Oxygen		0.06
Sulfur		0.004
Apparent density, g/cm^3:		2.85
Hall flow rate, s/50 g:		27
Screen analysis, wt%:		
+30 US Mesh	+600 µm	Trace
-30 +70	-600 +212	6
-70 +100	-212 +150	14
-100 +140	-150 +105	26
-140 +200	-105 +75	38
-200 +230	-75 +63	13
-230 +325	-63 +45	3
-325 US Mesh	-45 µm	Trace

Transverse rupture strength bars (TRS bars) and rings (5.26 cm OD, 4.34 cm ID and 0.635 cm thick) were pressed at 45 tsi (620 MPa) and 65°C using a dry film lubricant spray (based on Teflon™) to lubricate the die walls. The specimens were cured in air for 15, 30 and 60 minutes at seven different temperatures ranging from 150°C up to 500°C (150, 175, 200, 250, 300, 400 and 500°C). Three TRS bars and two rings were prepared for each curing time-temperature condition. The density was measured in water (Archimede's method). The electrical resistivity was evaluated using a four-point contact probe connected to a micro-ohmmeter and five readings were taken on top and bottom faces of each TRS bar and averaged. The transverse rupture strength tests were made according to MPIF Standard 41.

DC and low frequency AC measurements were carried out using an ACT/SMT-500 computer-automated magnetic hysteresisgraph. DC properties such as coercive field and maximum permeability were measured at an applied field of 150 Oe (11940 A/m). The DC hysteresis loss was also evaluated by numerical integration of the area of the DC loop. AC properties such as the relative maximum permeability and core loss were evaluated at 60 Hz and 400 Hz at a magnetization of up to 10 kG (1 T). The effect of the curing time (15, 30 and 60 minutes) on the magnetic properties is reported only for the 400°C and 500°C curing temperatures because it did not have a significant effect at a lower temperature.

RESULTS AND DISCUSSION

Mechanical and electrical properties

The effects of curing temperature and time on the mechanical strength of iron/0.8% resin composites pressed at 620 MPa (45 tsi) and 65°C are given in Figure 1. Note that the average density of the bars after curing was 7.20 ± 0.01 g/cm^3. The curing temperature significantly affects the mechanical strength of the samples. In the 150-250°C curing temperature range, the strength increases with time and seems to reach two plateaus. A first one appears at 150-175°C with a maximum strength of about 16,000 psi (110 MPa) and a second plateau at 200-250°C with a maximum strength of about 19,000 psi (130 MPa) which probably corresponds to a complete reticulation of the resin. At 300°C, the maximum strength is achieved in the first 15 minutes with a value of about 17,000 psi (120 MPa) and then decreases with time. Between 300°C and 500°C, the strength decreases continuously which likely corresponds to a deterioration of the resin and reaches a value of about 8,000 psi (55 MPa) at 500°C. Note that this value is nevertheless higher than the green strength of the composite material (before curing) which is about 3,500 psi (25 MPa).

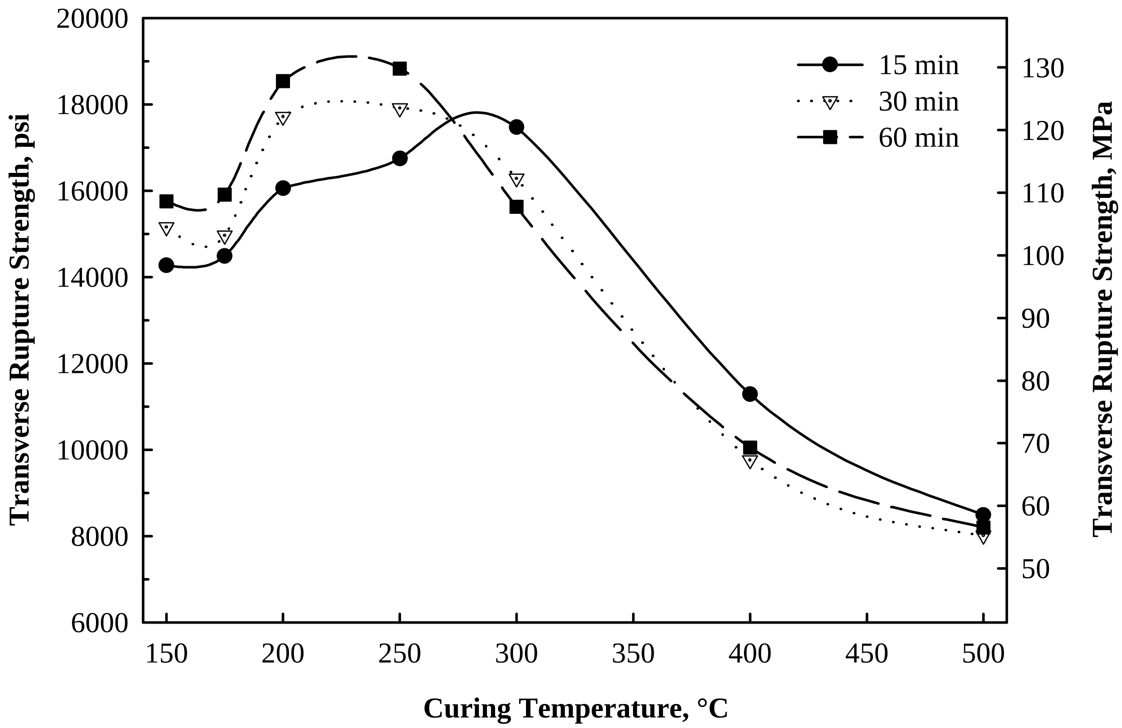

Figure 1. Effect of curing temperature and time on the mechanical strength of iron/0.8% resin composites pressed at 620 MPa (45 tsi) and 65°C.

The effects of curing temperature and time on the electrical resistivity are shown in Figure 2. As expected, the resistivity decreases rapidly with the curing temperature which is a more determinant factor than the curing time. After pressing and curing at 150°C, a resistivity of about 250 µohm-m is measured on the composites but it decreases continuously with an increase of the curing temperature down to about 2 µohm-m at 500°C. This is likely due to a deterioration of the resin and the formation of metallic

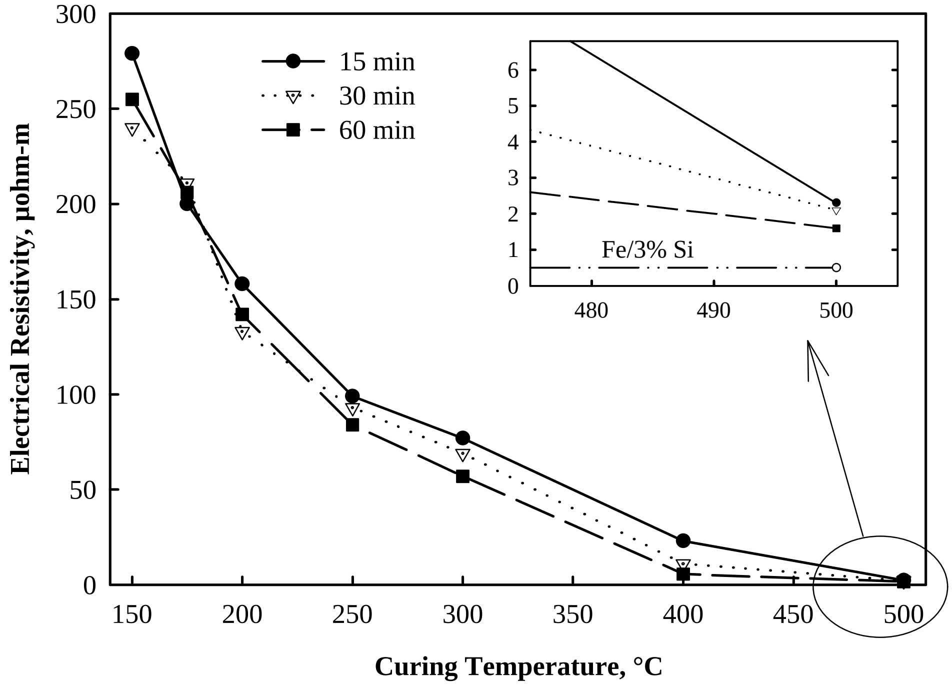

Figure 2. Effect of curing temperature and time on the electrical resistivity of iron/0.8% resin composites pressed at 620 MPa (45 tsi) and 65°C.

contacts between iron particles. However, as illustrated in the inset of Figure 2, the resistivity of an iron/3% silicon steel is about 0.5 µohm-m. This is still much lower than that of the composites after a curing treatment at 500°C.

A metallographic examination was carried out in order to illustrate the distribution of the insulation layer in these composites after curing. Optical micrographs of two composites cured in air at 175°C and 500°C for 30 minutes are shown in Figure 3. The micrographs look very similar. It seems that the temperature of the curing treatment did not have a significant effect on the distribution and thickness of the air gaps (insulation) between the iron particles. Thus the reason for the change in resistivity is not apparent on an optical micrograph.

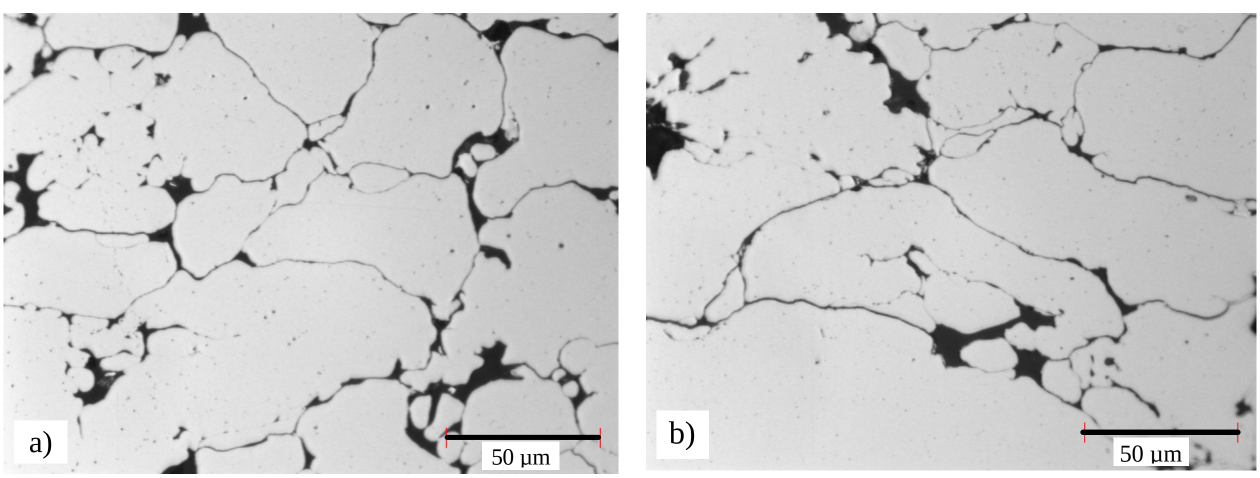

Figure 3. Optical micrographs of iron/0.8% resin composites pressed at 620 MPa (45 tsi) and 65°C and cured in air for 30 minutes at two temperatures: a) 175°C and b) 500°C.

DC magnetic properties

Although iron-resin composites are designed for AC magnetic applications, their DC characteristics are useful to better understand their magnetic behavior when subjected to an alternating magnetic field, especially at low frequency. The effect of the curing temperature on the shape of the magnetization loop at an applied field of 150 Oe (11940 A/m) is illustrated in Figure 4. The first half of the DC loop without the initial magnetization is shown for iron/0.8% resin composite rings cured 30 minutes in air at the lowest and highest curing temperatures used in this study: 150°C and 500°C. There is a significant change in the shape of the hysteresis curves with an increase of the slope of the curve at low field and a decrease of the area of the loop after a curing treatment at 500°C. In fact, these changes correspond respectively to an increase of the permeability μ and a decrease of the coercive field H_c.

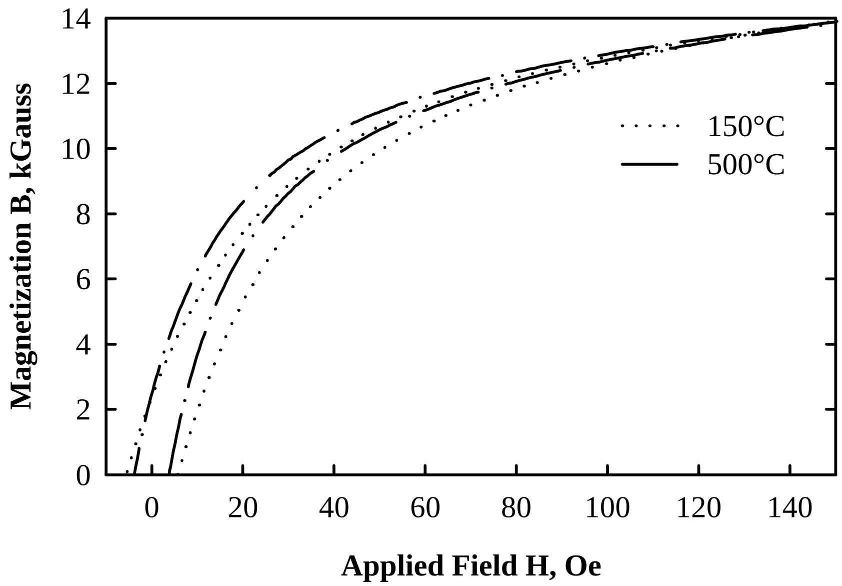

Figure 4. First half of the magnetization loop at an applied field of 150 Oe (11940 A/m) for iron/0.8% resin rings cured for 30 minutes at 150°C and 500°C.

The effects of the curing time and temperature on these two properties are shown in Figure 5. The coercive field (Fig. 5a) decreases with an increase of the curing temperature from 5.6 Oe (446 A/m) at 150°C down to 3.8 Oe (302 A/m) at 500°C. This represents a 30% decrease in coercivity. The curing time (15, 30 or 60 minutes) has little effect on the coercivity at 400°C and 500°C. The curing temperature also significantly affects the permeability of the material. The maximum permeability μ_{max} (Fig. 5b) is almost constant up to 300°C with an average value of 260 and then increases sharply with temperature up to a value of 400 at 500°C. This represents an increase of about 50% in maximum permeability and here again, the curing time (15, 30 or 60 minutes) has little effect on the permeability of composite rings cured at 400°C and 500°C.

This improvement in maximum permeability and coercivity translates directly in a decrease of the hysteresis loss. This latter has been evaluated by integrating the surface area of the DC hysteresis loop of each sample. The variation of the calculated hysteresis loss with respect to the curing time and temperature is shown in Figure 6. On average, the hysteresis loss decreases from 2200 J/m³ for a composite sample cured at 150°C down to 1700 J/m³ at 500°C which corresponds to a total decrease of 23% in hysteresis loss. The data points reported at a curing temperature of 400°C and 500°C indicate that the longer the curing time, the lower the hysteresis loss.

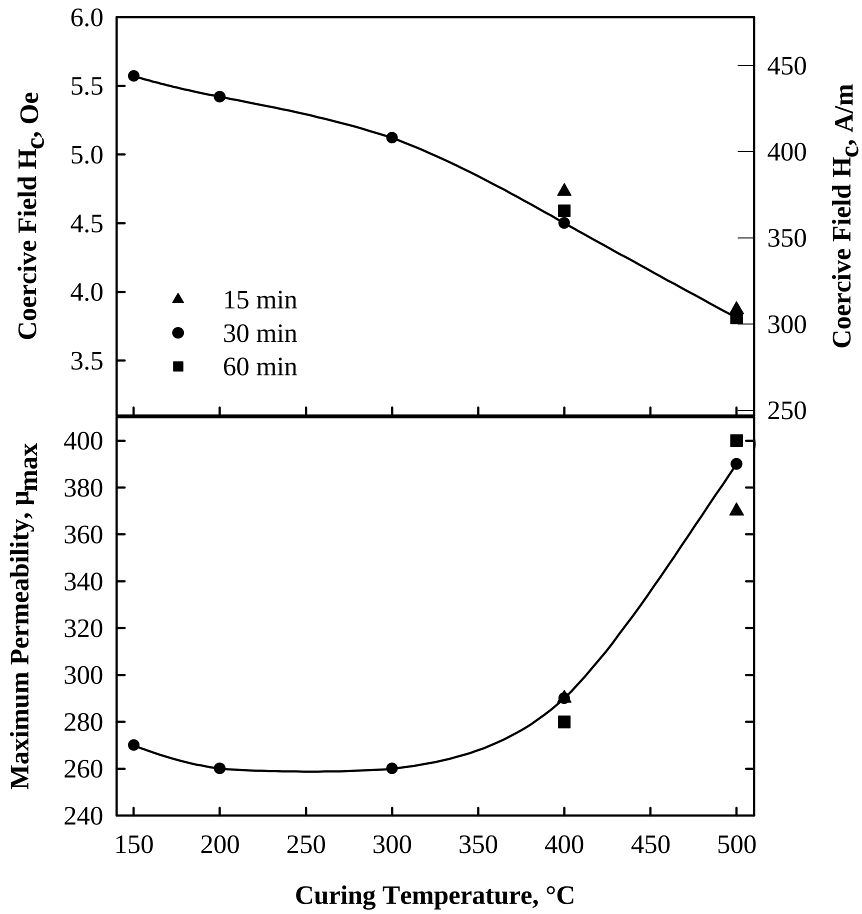

Figure 5. Effect of curing temperature and time on H_c and μ_{max} measured in a 150 Oe applied field (11940 A/m) for iron/0.8% resin composites pressed at 620 MPa (45 tsi) and 65°C.

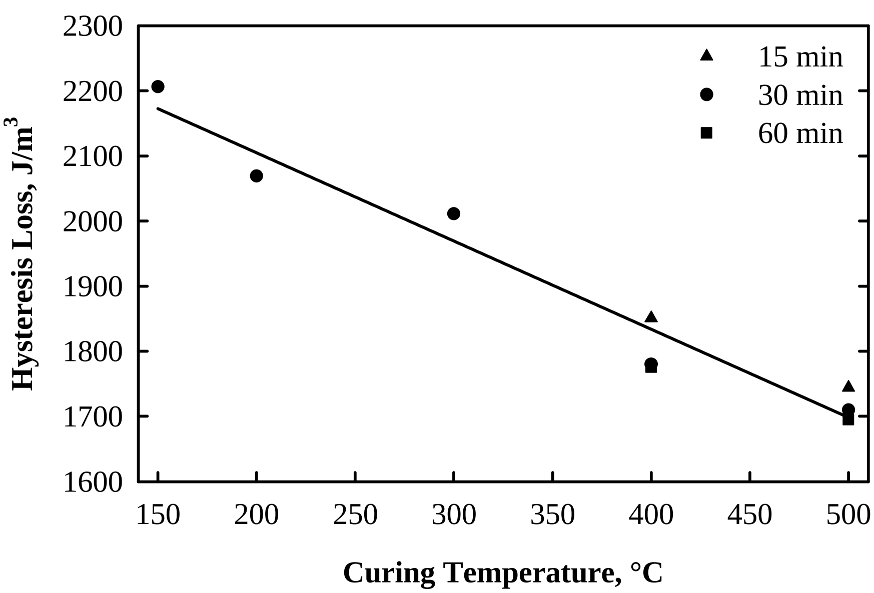

Figure 6. Effect of the curing time and temperature on the hysteresis loss of iron/0.8% resin composites pressed at 620 MPa (45 tsi) and 65°C.

AC magnetic properties

The effects of curing time and temperature on the core loss at 10 kG (1 T) and AC maximum permeability are shown in Figure 7 for iron/0.8% resin rings submitted to a varying field of 60 Hz. For a curing time of 30 minutes, there is a minimum in core loss at a curing temperature of 400°C with a value of 4.5 W/lb (10 W/kg) which represents a decrease of about 12% in core loss compared to the samples cured at the other temperatures. Time also has an effect on the core loss at a curing temperature of 400°C and 500°C. Indeed, at 400°C the minimum loss is obtained after 30 minutes while at 500°C the minimum is obtained after 15 minutes of curing. Therefore, a curing at 400°C for 30 minutes represents an optimum heat treatment with respect to the core loss at 60 Hz for this iron-resin system. It likely stress relieves the samples and decreases the hysteresis loss as reported in the preceding section while keeping a minimum value of electrical resistivity necessary to prevent the occurrence of eddy currents. At 500°C, the decrease in hysteresis loss resulting from the stress relief of the samples is canceled by the increase in eddy currents, the electrical resistivity being too low to shield the eddy currents.

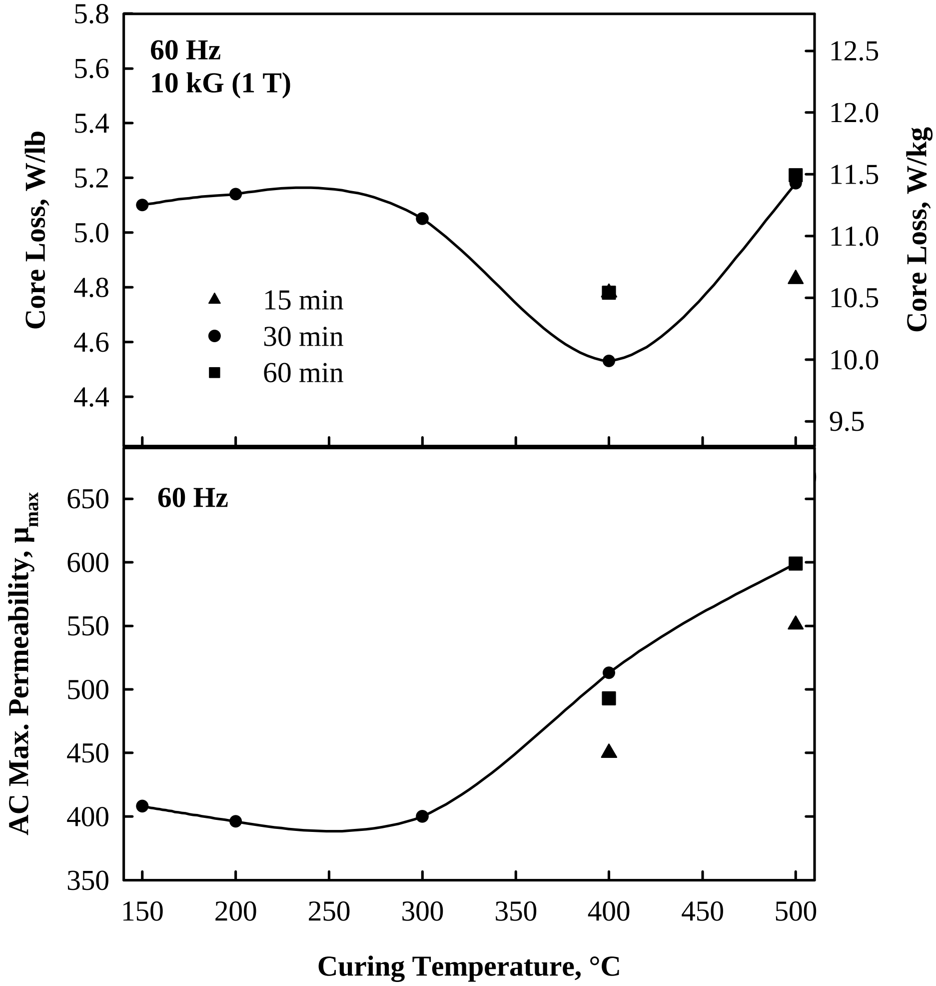

Figure 7. Effect of curing time and temperature on core loss at 10 kG (1 T) and AC maximum permeability at 60 Hz.

The effects of curing time and temperature on the AC maximum permeability are in accordance with the results obtained in the DC characterization. Indeed, the permeability increases from 400 at temperatures equal to or lower than 300°C up to 600 at 500°C which is still an indication of a stress relief. The curing time also has an effect at a curing temperature of 400°C and 500°C, the maximum permeability being slightly higher after 30 and 60 minutes compared with 15 minutes. It is worth noting that the maximum AC permeability at 60 Hz in these composites is obtained at an induction of about 5 kG (0.5 T).

The same core loss and AC maximum permeability measurements were carried out at 400 Hz. The results are presented in Figure 8. The core loss is constant up to about 400°C with an average value of 37 W/lb (80 W/kg) and then increases suddenly up to 86 W/lb (190 W/kg) for rings heat-treated at 500°C. It is interesting to note that the core loss is almost constant in the 150-400°C curing temperature range even if the electrical resistivity decreases from 250 µohm-m down to 20 µohm-m at 400°C. This is a good indication that the insulation in these composites is largely sufficient for applications in the 60-400 Hz range and beyond. However, a curing treatment at temperatures greater than 400°C results in a significant decrease in electrical resistivity and in an important increase of eddy currents at 400 Hz.

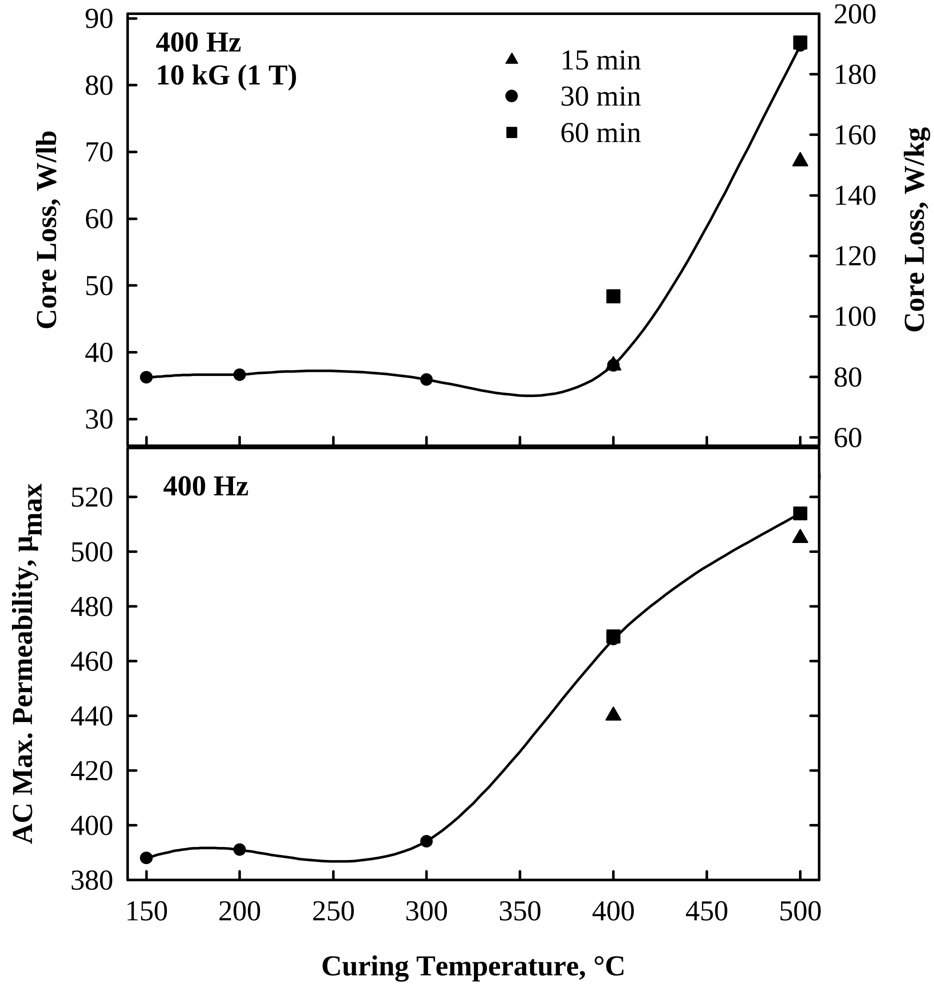

Figure 8. Effect of curing time and temperature on core loss at 10 kG (1 T) and AC maximum permeability at 400 Hz.

Also the AC maximum permeability increases with the curing temperature but the effect is less than that observed at 60 Hz: from 400 at temperatures equal to or less than 300°C up to 500 at 500°C. This smaller increase in permeability at 400 Hz is also an indication of important eddy currents in materials cured at 400-500°C. Thus there is no advantage to cure these iron-resin composites at high temperature for applications at 400 Hz because the decrease in hysteresis loss due to the stress relief is lower than the increase in losses due to eddy currents.

CONCLUSION

This study highlighted the beneficial effects of curing iron-resin composites at temperatures higher than usually recommended curing temperatures. It has been found that the highest mechanical strength is obtained by curing in the 200°C to 250°C range which yields composites with a high electrical resistivity and good magnetic properties in the 60 to 400 Hz range and beyond. For applications at 60 Hz, it is valuable to cure the composites at a higher temperature in order to decrease the hysteresis loss which accounts for about 95% of the total core loss at 60 Hz in this type of soft magnetic material [3]. By curing at 400°C for 30 minutes in air the magnetic properties at 60 Hz are optimized with a decrease of 19% in hysteresis loss which translates into a decrease of 12% in total core loss and an increase of 25% in AC permeability.

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