

EFFECT OF THERMAL CYCLING ON PROPERTIES OF SOFT MAGNETIC IRON-RESIN COMPOSITES

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ABSTRACT

Pressed and cured iron-resin composites have good mechanical and AC magnetic properties. Household appliances and magnetic components for the automotive industry are examples of potential applications. In real life, composites used in these applications are submitted to climatic and under-the-hood temperature variations. These ambient conditions may affect their performance.

A study has been conducted to evaluate the effect of thermal cycling on mechanical and magnetic properties of iron-resin composites. In this paper, the properties of pressed and cured iron/0.8% resin composites cycled from -40°C to 150°C are presented and discussed.

INTRODUCTION

Iron-resin composites are dielectromagnetics composed of ferromagnetic particles electrically insulated by an organic and/or inorganic dielectric material [1]. The idea of using iron-resin composites for soft magnetic applications is not new. It appeared more than 100 years ago but iron-resin composites have been rarely used because their properties, the processing technology for making parts and real needs for these materials were not sufficiently developed [2]. However these limitations are being overcome with the development of improved raw materials and new shaping technology.

For instance, new iron-resin composites have been recently developed and are now commercially available. The ease of consolidating such materials into complex shapes allows the manufacturing of net shape components for electrical micromotors and low power motors, which are extensively used for automation, robotics and office and home appliances. In addition, the increasing number of electromagnetic devices used in the automotive industry open new potential applications. Thus, it has become important to improve our knowledge on the behavior of these iron-resin composites under extreme operating environments in order to help designers in the integration of these new materials [3].

The environmental conditions from natural and vehicle-induced sources may influence the performance and reliability of automotive electronic and magnetic equipment. Among the environmental factors that can affect magnetic devices, there are temperature, humidity, atmosphere, dust, mechanical vibrations and shocks, and the general electrical environment. Thermal factors are probably the most omnipresent environmental hazards in automotive electronic and magnetic components. In the under-the-hood and indoor automotive applications, the ambient temperature of a magnetic device varies with time. These temperature variations may affect the properties of iron-resin composites, especially their magnetic properties [4] and mechanical strength.

For instance, it is known that the coercive force and hysteresis loss in iron decrease with an increase of the temperature while the permeability first increases and then decreases when temperature comes close to the Curie temperature [5]. The electrical resistivity of iron is also affected by temperature and almost doubles when temperature increases from 0°C up to 200°C [6]. This means that an increase of the temperature likely reduces the eddy currents induced by the AC magnetic fields and then the total loss. However, this effect is probably not as important in iron-resin composites at low frequency because total losses are mainly composed of hysteresis losses [7]. From a mechanical point of view, iron-resin composites exposed to temperature variations may be susceptible to failure or formation of cracks due to a mismatch in the thermal expansion coefficient of the resin and iron materials. It is also possible that a perfectly cured resin can, on cooling down from its curing temperature, develop cracks due to thermal shrinkage [8]. Under thermal cycling some parts of a magnetic device may expand more or less than others, causing cracks. These kinds of failures can be prevented by adequate care in processing and the choice of suitable resins. The insulated resin used in iron-resin composites should be selected in order to minimize the effect of temperature variations on magnetic and mechanical properties. Phenolic resins are materials of choice in a wide variety of heavy duty electrical applications since they have dielectric properties well suited for applications at service temperatures as high as 290°C [9]. In addition, phenolic resins show excellent resistance to weathering, are inherently flame-retardant, and are resistant to attack by most chemicals.

In this paper, laboratory test methods have been adapted to simulate the thermal conditions found in real-life environments and study their effects on an iron-0.8 wt% resin material developed for low frequency soft magnetic applications [10]. In particular, the effects of temperature and thermal cycling in the -40°C to 150°C range on mechanical and DC/AC magnetic properties are evaluated. The results of the study and principal characteristics of the material are herein presented.

MATERIAL DESCRIPTION

An iron-resin material composed of a screened (-30/+200 U.S. Mesh) high purity, high compressibility, water-atomized, iron powder and a phenolic resin has been used for the study. The choice of iron powder and the type and quantity of phenolic resin (0.8 wt%) was optimized in order to provide composite materials having excellent magnetic properties, high electrical resistivity and good mechanical strength. This iron-resin material system allows the use of a simple cost efficient processing technique for manufacturing: dry blending of the constituents and normal compaction followed by a curing treatment at low temperature. The selection of a thermoset instead of a thermoplastic was done to minimize the effect of the temperature variations on the magnetic and mechanical properties of the composites. Actually, the mechanical properties (modulus-temperature behavior) of thermosets are generally less affected by the temperature than thermoplastics [10].

EXPERIMENTAL PROCEDURE

Samples preparation and properties measurement

In this study, the iron/0.8% resin powder was shaped into rectangular bars for electrical and mechanical characterization and into rings for magnetic characterization. For each experimental condition, five bars measuring 3.175 cm long by 1.27 cm wide and 0.635 cm thick and five rings measuring 5.08 cm OD by 4.45 cm ID and 0.635 cm thick were consolidated. The specimens were uniaxially pressed in a double action floating die at 65°C under a compacting pressure of 620 MPa (45 tsi). Die walls were lubricated with a Teflon™ spray and the powder was preheated in the die during approximately two minutes prior to compaction. After compaction, all the specimens were cured in air at 175°C for one hour.

The density of each specimen was evaluated using Archimede's technique. The electrical resistivity was measured using a commercial micro-ohmmeter equipped with a four-point probe (0.8 cm between contact points). Ten resistivity measurements (five readings on top side and five on bottom side) were taken on each rectangular bar and averaged. The transverse rupture strength (TRS) was measured in accordance with MPIF Standard 41 on five bars for each experimental condition using a three-point bending system fixed to an Instron testing machine.

The magnetic properties were evaluated using a computer-automated magnetic hysteresisgraph. For DC magnetic characterization, five rings were wound with 600 primary turns of #24 gauge insulated copper wire and 150 secondary turns of 30 # gauge copper wire. The coercive force, residual induction, maximum permeability and maximum induction for an applied field of 11.9 kA/m (150 Oe) were evaluated. For AC characterization, five rings for each experimental condition were wound with 250 primary turns of #24 gauge copper wire and 250 secondary turns of #30 gauge copper wire. The AC apparent permeability and core loss were evaluated at an induction of 1.0 T (10 kG) at 60 Hz and 400 Hz together with the maximum apparent permeability which is usually obtained at a lower induction.

Thermal cycling: apparatus and experimental conditions

A special assembly was made to cool and warm the samples in a controlled environment and to be able to measure the magnetic properties in real time. The samples were placed in the thermal chamber shown in Figure 1 which is normally used with a mechanical testing machine (MTS) to measure mechanical properties at a specific temperature or during thermal cycling tests. The built-in temperature controller of the test chamber controls the heating, while for the cooling, the test chamber has been connected to a liquid nitrogen cylinder. The temperature inside the chamber is controlled by varying the opening of an electrical valve installed on the liquid nitrogen line at the rear of the unit. A thermocouple introduced through a hole on the top of the chamber and placed close to the specimens monitors the temperature and gives a feedback to the electric valve. Liquid nitrogen is admitted into the chamber and an electric fan insures a good uniformity of the temperature inside the chamber which was held within $\pm 2^\circ\text{C}$ of the target values during tests. The temperature was recorded throughout testing and the humidity was not controlled but recorded during the thermal cycles. Values of 44% and 0% relative humidity were measured at -40°C and 150°C respectively.



Figure 1: Thermal chamber assembly with the temperature programmer and recorder.

The composite rings were wound and suspended on two horizontal glass rods as shown in Figure 1, to insure a uniform exposure to temperature. The primary and secondary winding terminals of each ring were extended in order to exit the chamber via the door and connected directly to the hysteresigraph. Composite TRS bars were also placed in the test chamber in order to evaluate the effect of the number of thermal cycles between -40°C and 150°C on the transverse rupture strength.

The complete assembly with the thermal chamber and magnetic characterization system is shown in Figure 2. During thermal cycling, each ring was connected one at a time to the hysteresigraph for DC/AC magnetic characterization. The effect of temperature on magnetic properties (AC at 60 and 400 Hz and DC at 150 Oe) was evaluated at -40°C , 20°C and 150°C in real time during the first thermal cycle. The mechanical and magnetic properties were also evaluated at room temperature (20°C) after 1, 15 and 30 cycles.



Figure 2: Complete assembly with the thermal chamber and magnetic characterization system.

The thermal cycling used in the present study is similar to the one recommended for the environmental testing of electronic equipment in accordance with the SAE J1211 Standard. The selected thermal cycle profile selected is shown in Figure 3. This profile was selected because it permitted more temperature cycles for a given test duration. The maximum temperature selected is the one recommended for typical engine temperatures reached underhood. The transition rate (4°C/min) was the maximum ambient temperature transition rate suggested in the SAE J1211 standard. It allows the simulation of thermal shocks and its effects on the properties of the materials.

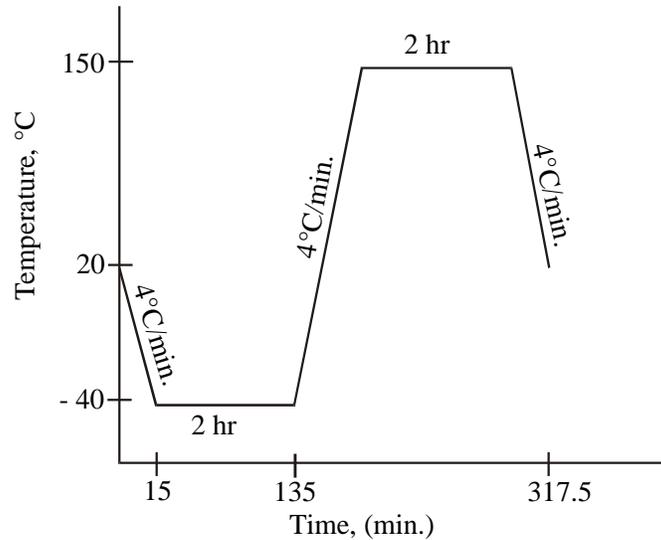


Figure 3: Thermal cycle profile used to simulate a thermal shock (according to SAE J1211 Standard).

RESULTS AND DISCUSSION

A-Properties of iron-0.8 wt% resin composites

The density and electrical resistivity measured before and after curing of iron-0.8% resin composites pressed at 65°C/620 MPa are presented in Table I. These properties are not significantly affected by the curing. The electrical resistivity gives an indication of the degree of insulation between iron particles. In comparison, the resistivity of pure water-atomized iron powder without resin pressed and cured in the same condition is typically 2 $\mu\Omega\text{-m}$ [7].

Table I. Average and standard deviation of the density and electrical resistivity of iron-0.8% resin composites (20 bars and 12 rings) pressed at 65°C/620 MPa before and after a curing treatment at 175°C/1h in air*.

Density		Electrical resistivity (3 bars)	
Before curing	After curing	Before curing	After curing
g/cm^3	g/cm^3	$\mu\Omega\text{-m}$	$\mu\Omega\text{-m}$
7.18 (0.03)	7.18 (0.03)	172 (28)	180 (28)

* all properties were evaluated at room temperature (20°C)

B-Effect of temperature on magnetic properties

- DC magnetic properties

The first half of the DC hysteresis loop at an applied field of 11.9 kA/m (150 Oe) for iron-0.8% resin composites measured at -40°C, room temperature and 150°C is shown in Figure 4 (measured during the first thermal cycle). The most apparent changes are in the coercive force and in the area of the hysteresis loop which decrease with an increase in temperature as shown in the inset. The area of the hysteresis loop corresponds to the hysteresis loss and can be calculated by integrating the B-H curve. These values were evaluated and are reported in Table II with other DC magnetic properties extracted from the curves. Four rings were used for the measurements at each temperature which permitted evaluation of the standard deviation for measured properties. This standard deviation takes into account not only the variation due to the characterization itself but also due to the material processing.

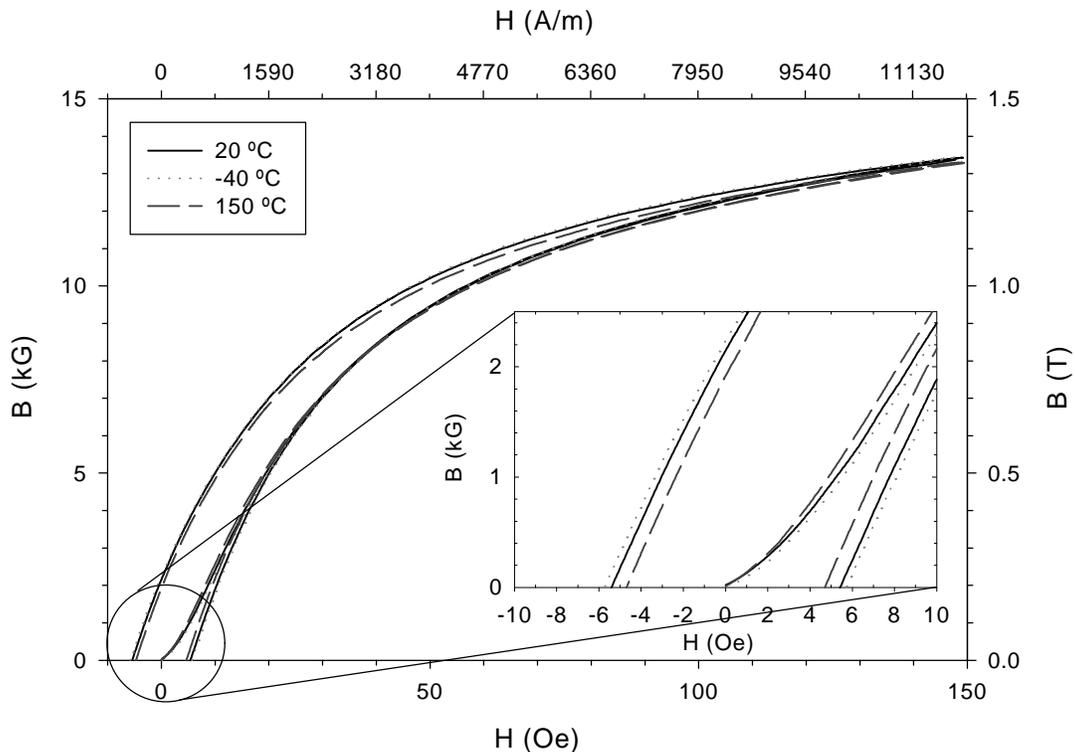


Figure 4. First half of the DC hysteresis loop of iron-0.8% resin composites at -40°C, 20°C and 150°C (rings pressed at 65°C/620 MPa and cured at 175°C/1 h in air).

Table II. Averages and (standard deviations) of DC magnetic properties of iron-0.8% resin composites measured at an applied field of 11.9 kA/m (150 Oe) at -40°C, 20°C and 150°C.

T	H _c	B _r	μ _{max}	B _s	Hysteresis loss
°C	A/m	T	T-m/A	T	J/m ³
-40	463 (4)	0.222 (0.014)	253 (11)	1.346 (0.005)	2187 (81)
20	430 (1)	0.212 (0.010)	254 (7)	1.341 (0.005)	2035 (41)
150	377 (4)	0.191 (0.008)	264 (7)	1.325 (0.007)	1743 (43)

The induction at saturation (B_s) and the maximum permeability (μ_{max}) are slightly affected by the temperature in the range studied. A decrease of less than 2% in magnetization (B_s) and an increase of 4% in maximum permeability (μ_{max}) were measured for an increase of the temperature from -40°C to 150°C. These results are in accordance with previous results obtained for samples submitted to smaller temperature variations [11]. It is interesting to note that the change in maximum permeability observed here is less important than that reported for a high purity electrolytic iron which gave an increase of about 80% in maximum permeability for a variation of the temperature from room temperature to 200°C [12].

The residual induction (B_r) and coercive force (H_c) are more affected by the temperature variation: a decrease of 18% in coercive force and 14% in residual induction for an increase of the temperature from -40°C to 150°C. The decrease in coercive force is again less than that reported for electrolytic iron where a decrease of 40% was observed for a similar temperature change [13]. It is known that for soft magnetic applications, it is more advantageous to have materials with low coercivity and residual induction in order to minimize the hysteresis loss which is proportional to the area of the DC loop. This is exactly what happened with these materials, as shown in Figure 5. The variations in DC properties and especially the decrease in coercivity with an increase of the temperature translated into a decrease of 20% in the hysteresis loss: from 2187 J/m³ at -40°C down to 1743 J/m³ at 150°C. This improvement in hysteresis loss is directly proportional to the decrease in coercive force. It is believed that this improvement is essentially due to a thermal activation which facilitates the movement of the magnetic domains or Bloch walls. The results indicate that the DC magnetic properties improve slightly with an increase of the temperature. This is in accordance with previous observations where magnetic field, stress and temperature were found to have a positive effect up to a certain level on the magnetization of iron [5].

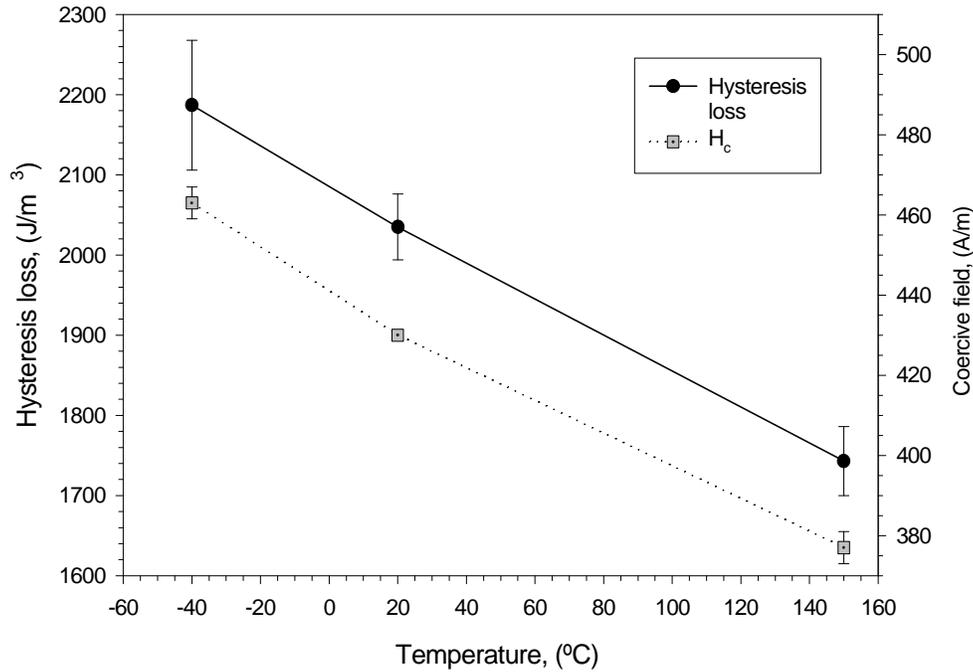
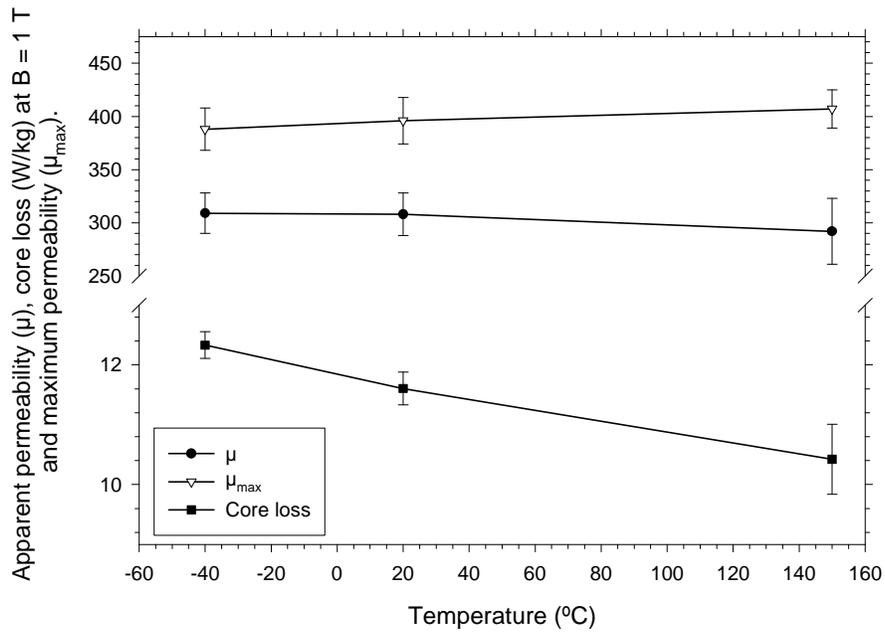


Figure 5. Variations of the hysteresis loss and coercive field with the temperature (-40°C, 20°C and 150°C) for iron-0.8% resin composite rings pressed at 65°C/620 MPa and cured at 175°C/1 h in air.

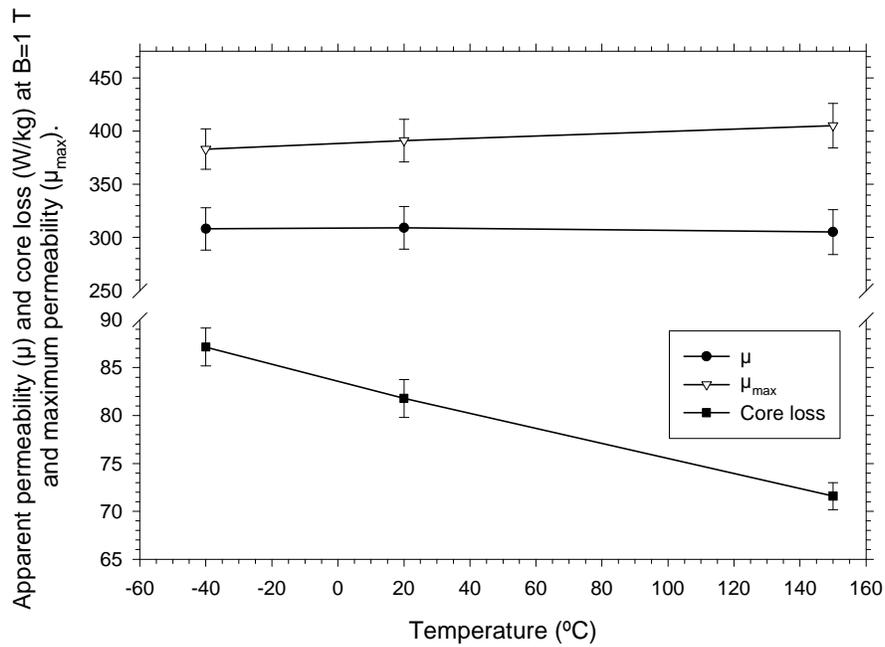
AC magnetic properties

The effect of temperature on AC magnetic properties at 60 Hz and 400 Hz for iron-0.8% resin composites pressed at 65°C/620 MPa and cured at 175°C/1 h in air is shown in Figure 6. The apparent permeability evaluated at a magnetization of 1 T (10 kG) and the maximum permeability are quite stable when the temperature increases from -40°C to 150°C: approximately 300 and 400 respectively (60 Hz or 400 Hz). In fact, the standard deviations reported on these curves indicate that there is no significant difference at 95% confidence interval in apparent or maximum permeability with the temperature change.

On the other hand, there is a significant decrease in core loss as temperature increases from -40°C to 150°C: 15% and 18% respectively at 60 Hz and 400 Hz. This improvement in core loss results partly from a decrease in hysteresis loss and partly from a decrease in eddy current loss. Indeed, it has been seen in the DC characterization that a decrease of 20% in hysteresis loss was observed when the temperature increases from -40°C up to 150°C. Also, it is known that the electrical resistivity of iron doubles when the temperature increases from 20°C up to 200°C [6]. This increase in resistivity reduces the eddy currents and thus contribute to reduce the total core loss. This is especially true at 400 Hz (see Figure 6b) since the proportion of eddy currents in the total core loss increases with frequency.



(a) 60 Hz



(b) 400 Hz

Figure 6. Effect of temperature (-40°C , 20°C and 150°C) on the AC magnetic properties of iron-0.8 % resin composites pressed at $65^{\circ}\text{C}/620$ MPa and cured at $175^{\circ}\text{C}/1$ h in air. (a) 60 Hz, (b) 400 Hz

Mechanical property

The effect of the number of thermal cycles on the transversal rupture strength (TRS) of iron-0.8% resin composites is presented in Figure 7. The TRS increased from 105 MPa (15,500 psi) up to a constant value of 135 MPa (19,500 psi) after 15 cycles. However, due to the sampling used it is not possible to precisely determine the minimum number of cycles to reach the plateau shown in this Figure. The increase in strength is likely due to a completion of the curing of the resin or a modification of the interaction between the surface of the iron particles and the resin during the thermal cycling, and especially during the pauses in the cycle at 150°C (see Figure 3). Normally, the highest strength with these iron-resin composites is obtained after a curing in the 200°C to 250°C temperature range. Since the curing had been made at 175°C in the present case, it is possible that it continued at 150°C during the long pauses.

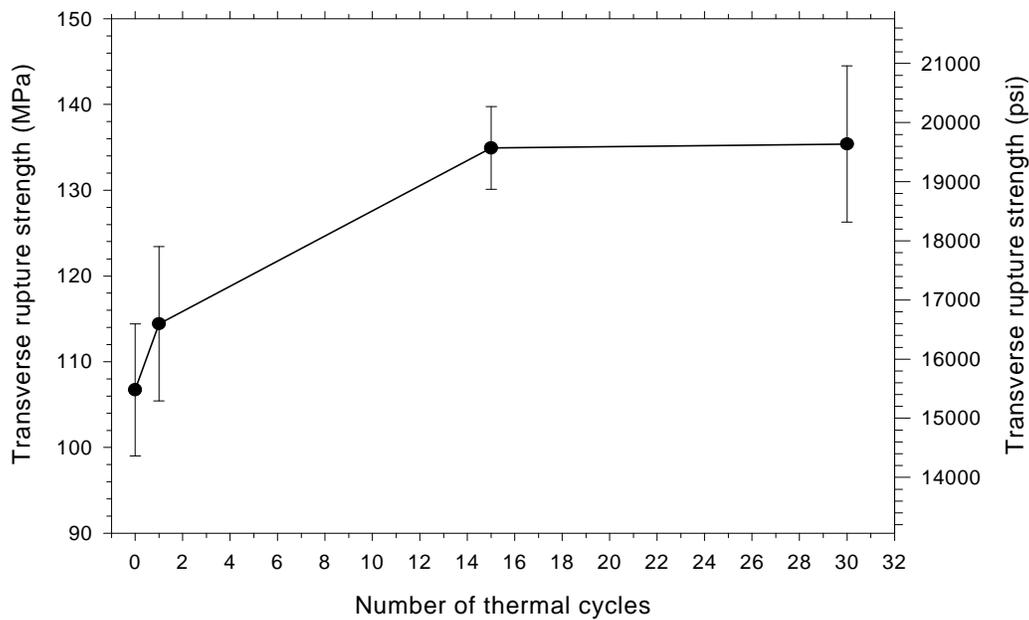


Figure 7: Effect of the number of thermal cycles on the transverse rupture strength of iron-0.8% resin composites pressed at 65°C/620 MPa and cured at 175°C/1 h in air. (Measured at 20°C on 5 bars per data point)

DC magnetic properties

The effect of the number of thermal cycles (-40°C to 150°C) on the DC magnetic properties of iron-0.8% resin composites pressed at 65°C/620 MPa and cured at 175°C/1 h is presented in Figure 8. The DC properties are very stable up to 30 thermal cycles. The largest change is observed for the coercive field which decreases by 10 A/m corresponding to a change of less than 2%. The DC properties are so stable that the standard deviation bars could not be shown on the graphs. For example, in the case of the saturation induction B_s , the standard deviations evaluated on 4 different rings are 0.005, 0.007, 0.004 and 0.008 T after 0, 1, 15 and 30 thermal cycles, respectively.

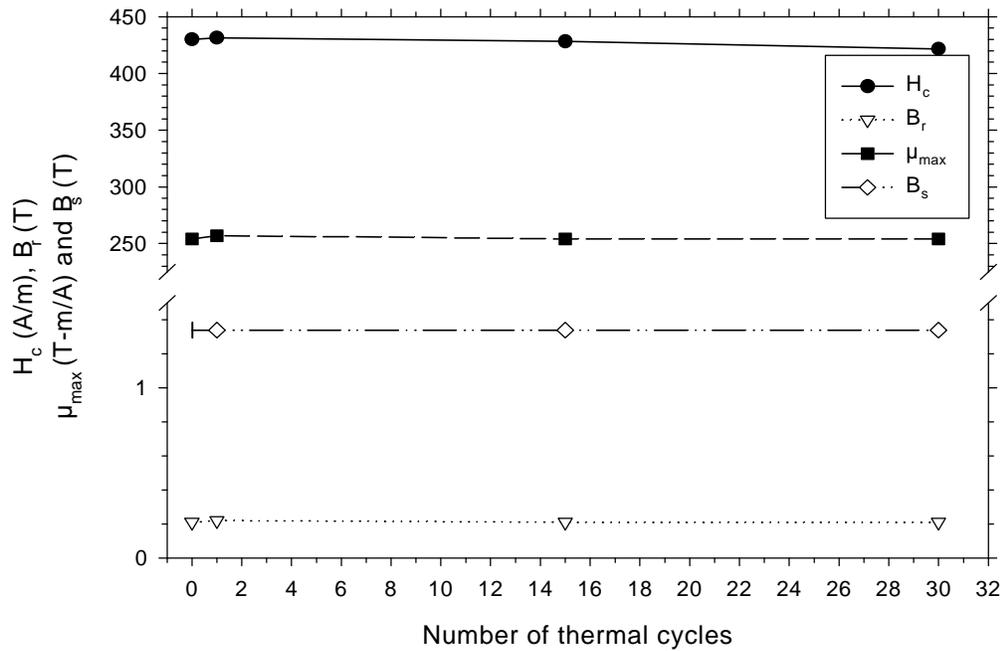
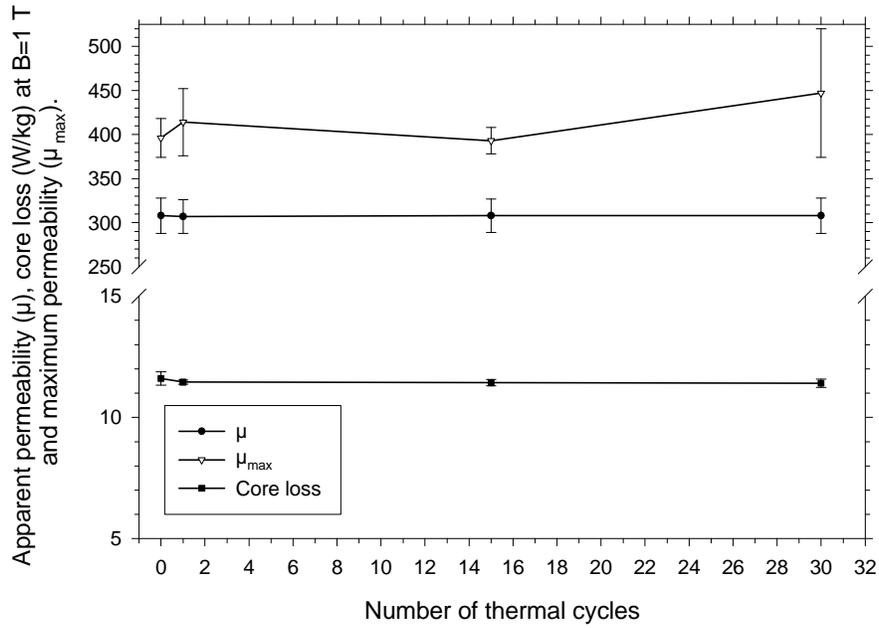


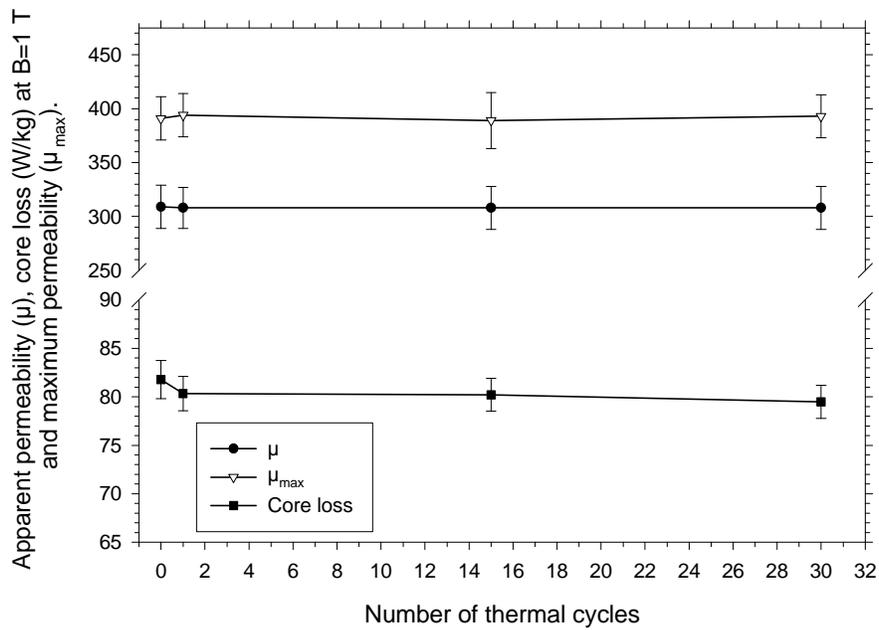
Figure 8: Effect of the number of thermal cycles (0, 1, 15 and 30) on DC magnetic properties of iron-0.8 % resin composites pressed at 65°C/620 MPa and cured at 175°C/1 h in air.

AC magnetic properties

The effect of the number of thermal cycles on the AC magnetic properties evaluated at 60 Hz and 400 Hz is shown in Figure 9. The AC magnetic properties are not significantly affected by the number of thermal cycles. A statistical analysis of the results at 60 Hz and 400 Hz showed that there is no significant difference at 95% confidence interval between the AC magnetic properties measured after the first cycle and after the 30th cycle. Furthermore, the standard deviations reported on the curves are mostly attributable to differences between rings and not to the precision of the hysteresisgraph. In fact, the highest standard deviations obtained at 400 Hz for a particular ring measured 4 times (0, 1, 15 and 30 cycles) are: 1.0 W/kg for the core loss, 0.6 for the apparent permeability and 5 for the maximum permeability.



(a) 60 Hz



(b) 400 Hz

Figure 9: Effect of the number of thermal cycles (0, 1, 15 and 30) on AC magnetic properties of iron-0.8% resin composites pressed at 65°C/620 MPa and cured at 175°C/1 h in air. a) 60 Hz, b) 400 Hz.

CONCLUSION

In this study the effect of temperature and a thermal cycling on the mechanical, DC and AC magnetic properties of iron-0.8% resin composites were evaluated. The thermal cycling from -40°C up to 150°C reproduced the extreme thermal conditions expected for electronic devices in automotive applications. The most important observations were:

- DC magnetic properties improve with an increase in temperature: the coercive force, residual induction and hysteresis loss decrease by 18%, 14% and 20% respectively.
- AC permeability evaluated at 60 Hz and 400 Hz are not affected by an increase of the temperature from -40°C up to 150°C while core losses decrease by 15% and 18% at the same frequency respectively.
- Strength of the iron-0.8% resin composites increased with the number of thermal cycles. A value of 135 MPa (19,500 psi) was obtained after 15 thermal cycles which is attributable to a better interaction between the surface of iron particles and the resin and to modifications of the resin properties.
- DC and AC magnetic properties evaluated at 60 Hz and 400 Hz do not vary with a 95% confidence interval with the number of thermal cycles.

Thus, the study showed that iron-0.8% resin composites are adequate materials for use in applications where the magnetic devices are submitted to climatic or under-the-hood temperature variations. Their properties are stable under thermal cycling and improves slightly with an increase of the temperature from -40°C up to 150°C.

ACKNOWLEDGMENT

The authors are extremely grateful to Paul-Émile Mongeon and Gilles St-Amand for their precious help and their technical skills.

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