RESIN IMPREGNATION OF SOFT MAGNETIC MATERIALS FOR LOW FREQUENCY APPLICATIONS

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ABSTRACT

Novel P/M techniques offer an economic alternative to produce net shape iron-resin composites with isotropic properties for AC magnetic applications. However, in order to use these materials at low frequencies, e.g. around 60 Hz, it is suitable to increase the permeability. Previous works have shown that the resin content of such materials should be kept as low as possible in order to increase permeability. Resin impregnation allows to significantly increase the interparticle bonding using a minimum amount of resin. This technique is an attractive way to provide parts with good mechanical strength, high permeability and low losses at low frequency. Results of mechanical and low frequency magnetic properties of iron compacts impregnated at different temperatures and pressures with different resins are presented and discussed.

1. INTRODUCTION

Powder metallurgy (P/M) technology is a well-known economic process for mass production of near-net-shape parts. This technique has been succesfully used to manufacture soft magnetic components intended for DC magnetic applications. These components are usually produced by compacting and sintering iron-based powder mixes (pure iron, iron-nickel, iron-phosphorus, etc.). For AC magnetic applications, soft magnetic iron powder composites or dielectromagnetics may be used [1]. These materials are essentially composed of high purity iron particles insulated from each other by a dielectric material. The purity and high compressibility of the iron powder insure good ferromagnetic properties while the dielectric material provides the mechanical properties and the required insulation between the iron particles that minimizes the magnetic losses. The dielectric material can be selected from organics, such as polymers or from inorganics, such as oxides, phosphates, silicates, etc. [2].

Dielectromagnetics exhibit a level of magnetic properties and a degree of technology that makes them suitable for new designs of magnetic components. They possess a relatively high degree of isotropic magnetic and thermal properties. Furthermore, at frequencies higher than 100 Hz, these materials have a relatively stable AC apparent permeability and low loss. However, for applications at very low frequencies (typically 50-60 Hz), the AC apparent permeability and core loss must be improved. The unsaturated permeability of these materials is relatively low and sometimes seen as an important drawback [3]. For example, typical AC permeability values range from 200 to 600 [4]. In order to improve these properties, the use of pure iron powder without any additive has been considered in which case higher density and permeability have been obtained, as expected [5].

Dielectrics generally used in the fabrication of iron magnetic composites can be admixed to the iron powder using a wet or dry mixing process [6] or they can also be applied as a thin coating on the iron particles (encapsulation) using a fluidized bed technique [7,8]. Iron powder parts can also be impregnated after compaction with a dielectric. The impregnation involves filling of the pores of a porous body with a non-metallic compound such as oil, wax or resin. The impregnation sealant penetrates into pores by capillary forces, external pressure or vacuum. Resin impregnation is generally used to seal the porosity in order to provide parts suitable for painting, plating and other metal finishing operations. Resin impregnation can also enhance mechanical properties and corrosion resistance. For instance, it can increase the mechanical strength, the impact toughness, the hardness, etc.

In the case of pure iron magnetic components containing no additive, resin impregnation was considered in order to increase the mechanical strength of the parts without affecting the magnetic properties. Pure iron specimens were pressed at two different temperatures and the effects of the impregnation parameters, such as temperature, pressure and type of resin, on mechanical and magnetic properties were evaluated. These resin-impregnated iron components are intended for low frequency applications.

2. EXPERIMENTAL PROCEDURE

In these experiments a high purity water-atomized iron powder (ATOMET 1001HP) without any lubricant or additive was used. Rectangular bars and rings were prepared by uniaxial warm compaction. They were pressed at 620 MPa (45 tsi) at 65°C and 120°C. For each experimental condition, a minimum of three transverse rupture strength (TRS) bars and two rings (5.08 cm OD x 4.45 cm ID x 1.27 cm thick) were compacted in a double action floating compaction die. Die wall lubrication with graphite spray was used for all experiments. The powder was preheated in the die prior to pressing. For each compaction temperature, samples (bars and rings) were impregnated with a solution of phenolic resin dissolved in acetone. Two levels of pressure and temperature of impregnation were evaluated. The detailed plan of experiments is described in Table I. For this series of experiments, pressure impregnation was preferred over vacuum impregnation in order to avoid any solvent evaporation. After impregnation, all the samples were cured in air at 175°C for 1 hour.

Table 1. Experimental plan used to study the impregnation of iron compacts with a solution of phenolic resin in acetone.

$T_{Compaction}$	°C	65				120			
$T_{Impregnation}$	°C	20		65		20		65	
$P_{Impregnation}$	MPa	1.38	13.8	1.38	13.8	1.38	13.8	1.38	13.8
	psi	200	2000	200	2000	200	2000	200	2000

A second series of experiments was carried out in which iron compacts were impregnated with various ready-to-use epoxy resins supplied by different manufacturers. Mechanical vacuum impregnation was used with these resins. The impregnated samples were cured following the procedure suggested by the manufacturers in order to fully cross-link the resin and increase the mechanical strength of the samples.

Density was calculated from the weight and physical dimensions of the parts and was evaluated only before the impregnation, i.e. on green parts. The electrical resistivity was measured before and after the impregnation and curing treatment using a commercial micro-ohmmeter equipped with a four-point probe (0.8 cm between contact points). Ten resistivity measurements were taken on each rectangular bar. The resistivities were averaged: five readings on top side and five on bottom side. The surfaces were slightly polished after impregnation and curing in order to remove the resin coating. In accordance with MPIF standard 41, the transverse rupture strength (TRS) was measured on each bar sample using a three point bending system fixed to an Instron testing machine.

The AC apparent permeability and apparent core loss at an applied field of 0.5 T (5 kG) were evaluated at 60 Hz using a SMT/ACT-500 computer-automated magnetic hysteresisgraph. Each ring was wound with 250 primary turns of #24 gauge insulated copper wire and 250 secondary turns of #30 gauge copper wire.

3. RESULTS AND DISCUSSION

3.1 Physical, electrical and mechanical properties

The effect of the compaction temperature on green density of TRS bars pressed at 620 MPa (45 tsi) before impregnation and curing is presented in Figure 1. As expected, the density increases with the compaction temperature. This phenomenon observed in warm compaction is related to an increase of the ductility of the material and a reduction of its strain hardening, thus favoring plastic deformation of particles during compaction [9]. An increase in green density from 7.26 to 7.29 g/cm^3 was obtained by increasing the compaction temperature from 65 to 120°C. Densities after impregnation and curing were not evaluated because sample surfaces were irregularly coated with phenolic resin.

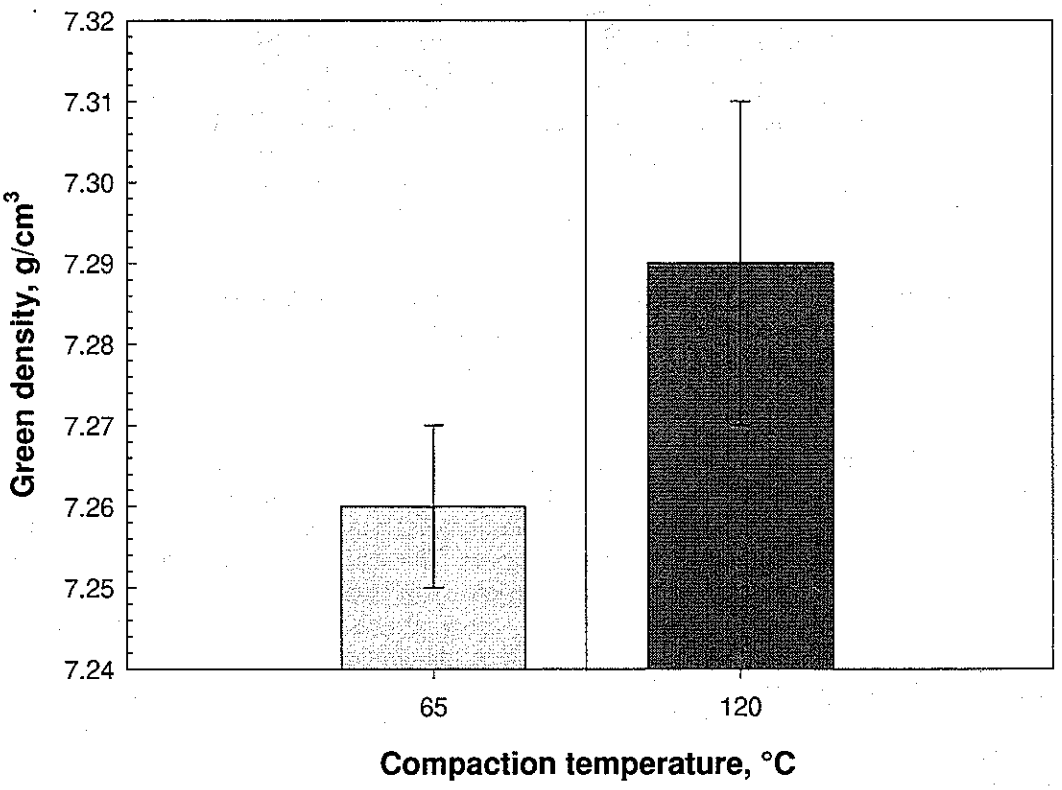


Figure 1: Effect of compaction temperature on green density of TRS bars pressed at 620 MPa (45 tsi).

The electrical resistivity before and after impregnation/curing of samples impregnated with phenolic resin as a function of the compaction temperature and impregnation parameters (temperature and pressure) is presented in Figure 2. The electrical resistivity of non-impregnated rectangular bars pressed at 620 MPa

(45 tsi) and 65°C before and after a similar curing treatment is also presented for comparison. As shown, the electrical resistivity of impregnated samples is similar to that of non-impregnated samples before curing but is approximately 50% higher after curing. A drop in resistivity is also observed after curing that is likely due to the formation of electrical contacts during curing. However, the presence of resin in impregnated samples partly prevents the formation of these contacts and produces samples with a higher resistivity.

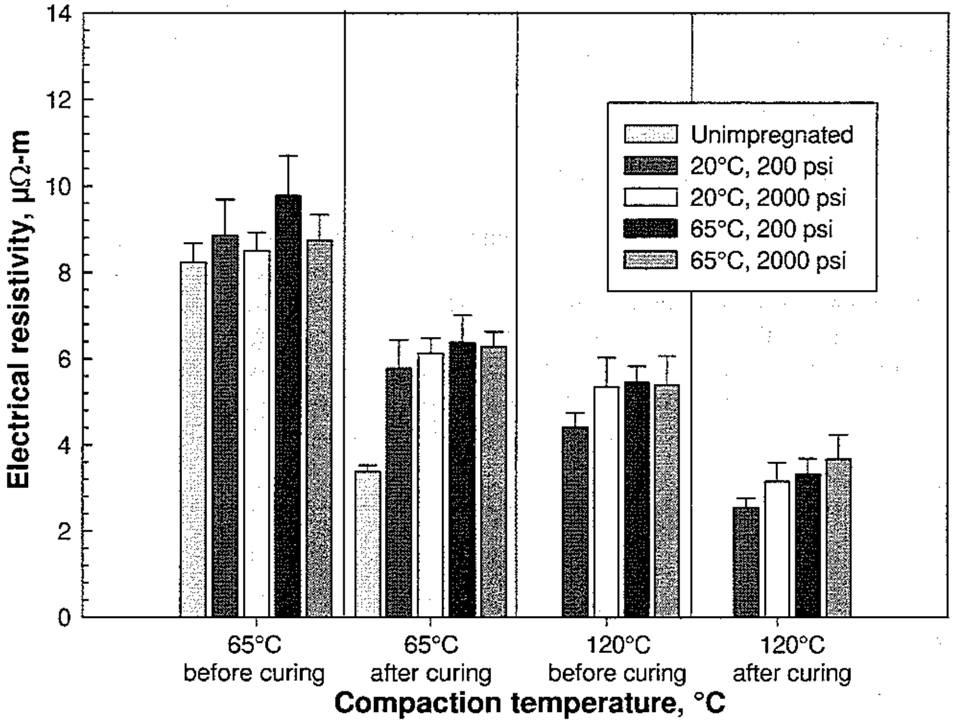


Figure 2: Effect of compaction temperature and curing treatment on electrical resistivity of specimens pressed at 620 MPa (45 tsi) and submitted to various impregnation conditions.

A statistical analysis of the results showed that the impregnation parameters (temperature and pressure) did not significantly affect the electrical resistivity of samples impregnated with phenolic resin at a 95% confidence level. On the other hand, the compaction temperature has a significant effect on electrical resistivity which decreases as the compaction temperature rises. This can be related to an increase of the number of electrical contacts between iron particles as the density of iron compacts increases. During the compaction process electrical contacts are created between iron particles leading to a resistivity drop. Thus, both the curing treatment and an increase of the compaction temperature cause a drop in resistivity. For instance, a 30% to 40% drop in resistivity is observed in samples during the curing treatment: from about 9 to 6 μohm-m for bars pressed at 65°C and from about 5 to 3 μohm-m for bars pressed at 120°C. It is interesting to note that the resistivity of the pressed samples after curing is nevertheless 30 to 60 times higher than that of pure wrought iron (~ 0.1μohm-m).

It is believed that during compaction, contacts are created between iron particles but, at the ejection stage, most of them separate from each other creating a thin distributed air gap and a relatively high resistivity iron compact. However, it has been observed that during the heat treatment in air, electrical contacts are created between iron particles and decreased the resistivity. These contacts are not simply or necessarily iron-to-iron contacts. Indeed, it has been showed that Fe_2O_3 (hematite) necks formed between iron particles during these heat treatments at low temperature [10]. The electrical resistivity of hematite being lower than that of air, it contributes to decrease the overall resistivity of the cured samples. The original distributed air gap is also filled by the phenolic resin during impregnation due to the capillary forces. The resin being more conductive than air it contributes to further decrease the overall resistivity.

The TRS of samples impregnated with phenolic resin and cured is presented in Figure 3 as a function of the compaction temperature and impregnation parameters (temperature and pressure of impregnation). The TRS of non-impregnated rectangular bars pressed at 620 MPa and 65°C and heat treated in air at 175°C for one hour is also presented for comparison. An impressive TRS value of approximately 57 MPa (8250 psi) was obtained for the non-impregnated samples. It is believed that this relatively high TRS value is due to the formation of oxide necks (hematite) between iron particles during the curing treatment. Figure 3 also shows that resin impregnation further increases the strength to approximately 92 MPa (13,300 psi) for samples pressed at 65°C and 99 MPa (14,300 psi) for samples pressed at 120°C. In the case of impregnated samples the improvement in TRS may be attributed to two concurrent mechanisms: the polymerization of the resin itself and the formation of oxide necks during curing.

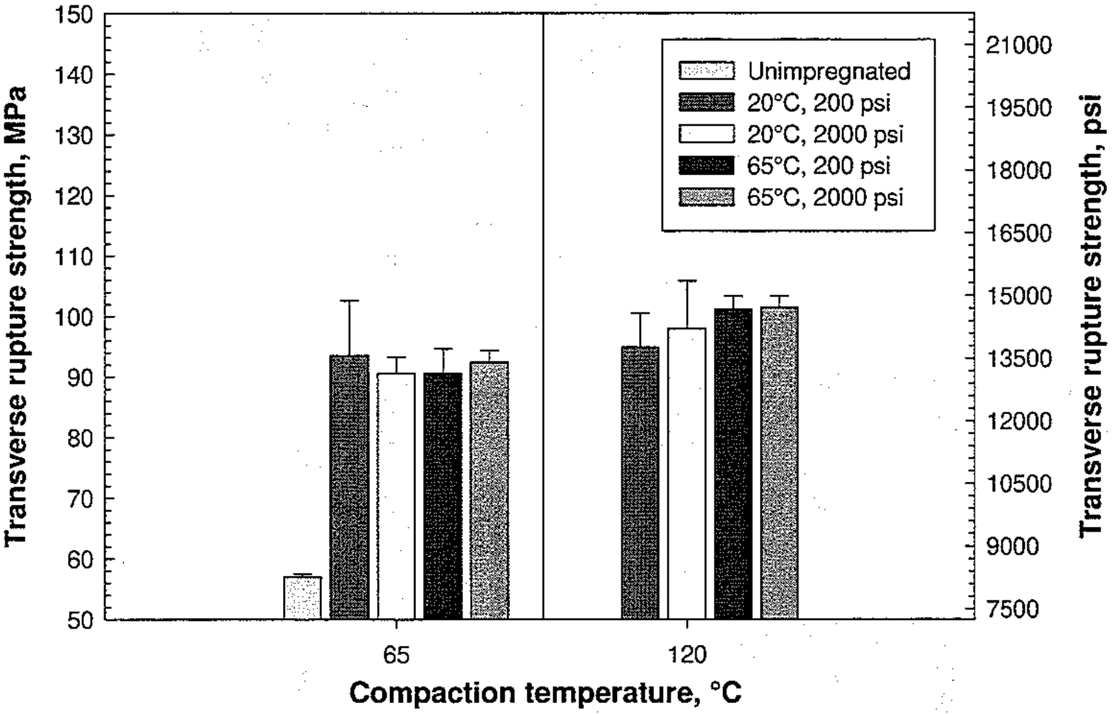
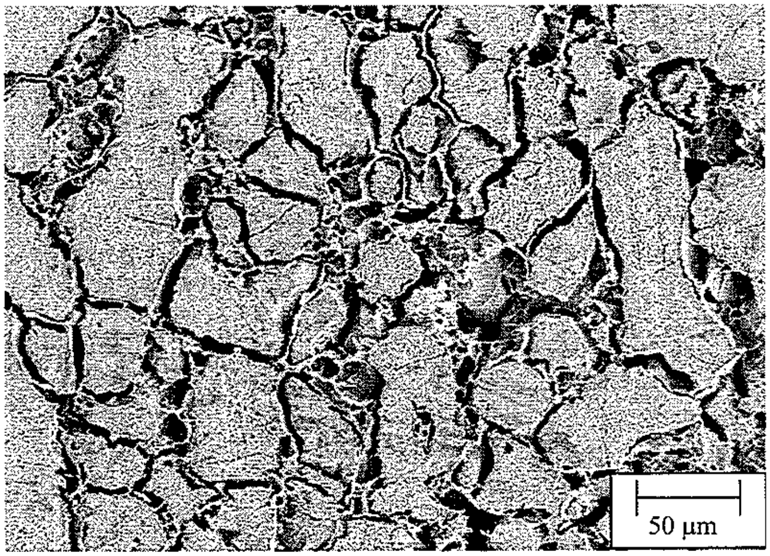
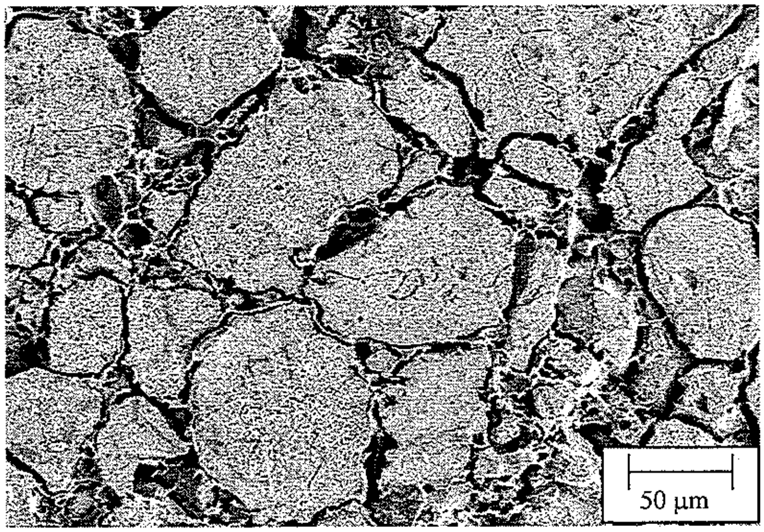


Figure 3: Effect of compaction temperature and impregnation parameters on TRS of bars pressed at 620 MPa (45 tsi) after curing in air at 175°C/1 h.

There is an increase of approximately 7 MPa (~1000 psi) in the TRS of impregnated samples by increasing the compacting temperature from 65°C up to 120°C. However, here again, a statistical analysis of the results showed that the impregnation parameters (temperature and pressure) did not significantly affect the TRS of samples impregnated with phenolic resin at a 95% confidence level. The pressure and temperature of impregnation selected in this study had practically no effect on the TRS probably because the impregnation solution (phenolic resin dissolved in acetone) had a low viscosity at room temperature and normal pressure. A metallographic observation confirmed that the resin completely impregnated the samples. Scanning electron micrographs (SEM), presented in Figure 4, show cross sections of two TRS bars after impregnation, curing, polishing and etching. Even if very different pressing and impregnation parameters were used for both specimens, the two micrographs show that the resin is well distributed around the iron particles forming a thin coating and filled all the porosity. Other experiments also confirmed that TRS values close to those reported here are achieved with iron compacts impregnated with the same phenolic resin at ambiant pressure and room temperature.



4a - TRS bars pressed at 65°C and impregnated at 65°C and 2000 psi.



4b - TRS bars pressed at 120°C and impregnated at 20°C and 200 psi.

Figure 4: Distribution of the resin in the center of TRS bars Impregnated with a phenolic resin and cured 1 h at 175°C in air (after polishing and etching). The etchant partly dissolved the iron powder and allowed to reveal the resin network.

3.2 AC magnetic properties at low frequencies

Core loss after curing (@ 60 Hz, 0.5 T) of non-impregnated and impregnated rings with the phenolic resin is presented in Figure 5 as a function of the compaction temperature and impregnation parameters (temperature and pressure). At 60 Hz, the core loss is not affected by impregnation. However, core loss slightly increases with the compaction temperature. In the case of impregnated samples, this can be related to decreasing resistivity as the compaction temperature rises (see Figure 2) thus increasing eddy current losses. For unimpregnated samples compacted at 65°C, an unexpected low value of core loss was obtained. Indeed, based strictly on the electrical resistivity of these samples that is similar to those measured on impregnated samples compacted at 120°C, a higher core loss value was expected. Additional experiments should be done to clarify this discrepancy.

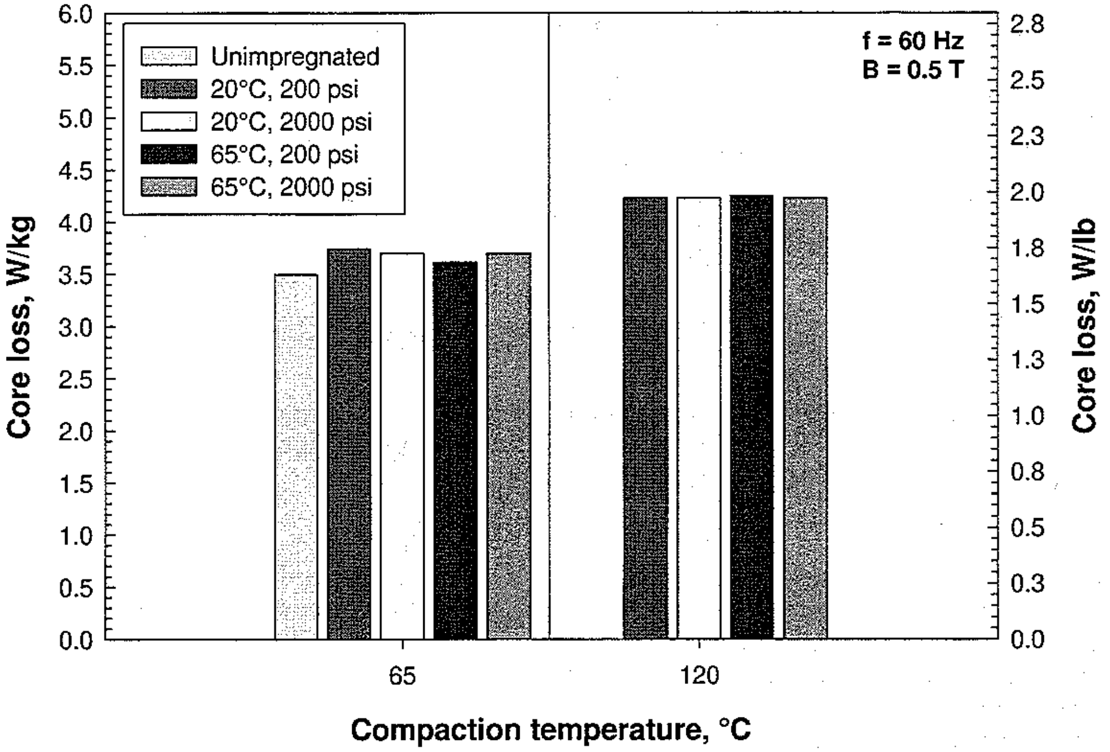


Figure 5: Effect of compaction temperature and impregnation parameters on core loss of rings pressed at 620 MPa (45 tsi) and cured 1 hour at 175°C in air.

AC apparent permeability after curing (@ 60 Hz, 0.5T) of non-impregnated rings and rings impregnated with the phenolic resin is presented in Figure 6 as a function of the compaction temperature and impregnation parameters (temperature and pressure of impregnation). At 60 Hz, the relatively high permeability of approximately 750 that is obtained with these samples is not affected by the impregnation. However, it slightly decreases when compaction temperature is increased to 120°C. This can be related to the resistivity of the samples which decreases as the compaction temperature rises leading to an increase of eddy currents. However, the differences observed are small and may be unsignificant.

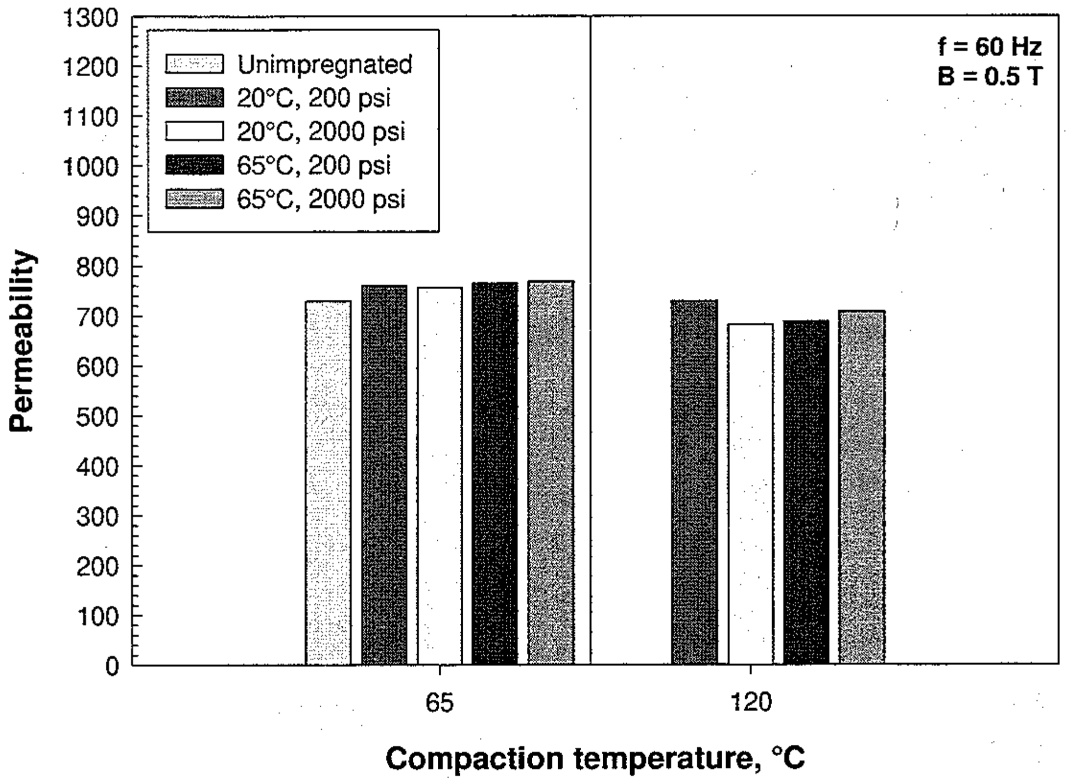


Figure 6: Effect of compaction temperature and impregnation parameters on apparent permeability of rings pressed at 620 MPa (45 tsi) and cured 1 hour at 175°C in air.

The following results refer to iron specimens impregnated under vacuum with three different ready-to-use epoxy resins. Figure 7 presents strength and AC magnetic properties measured on iron compacts impregnated and cured. These results show that the resin composition has a great impact on the mechanical strength. TRS values varying from 55 MPa up to 120 MPa (8000 to 17,500 psi) can be obtained depending on the composition of the resin. The highest TRS value obtained with the third epoxy resin corresponds to an increase of about 30% with respect to the previous results reported for the phenolic resin (see Figure 3). Furthermore, this increase in strength is achieved without affecting the AC magnetic properties. Indeed, apparent permeability as high and core loss as low as those achieved with non-impregnated samples were obtained @ 60 Hz and 0.5 T: approximately 760 in apparent permeability and 3.45 W/kg in apparent core loss.

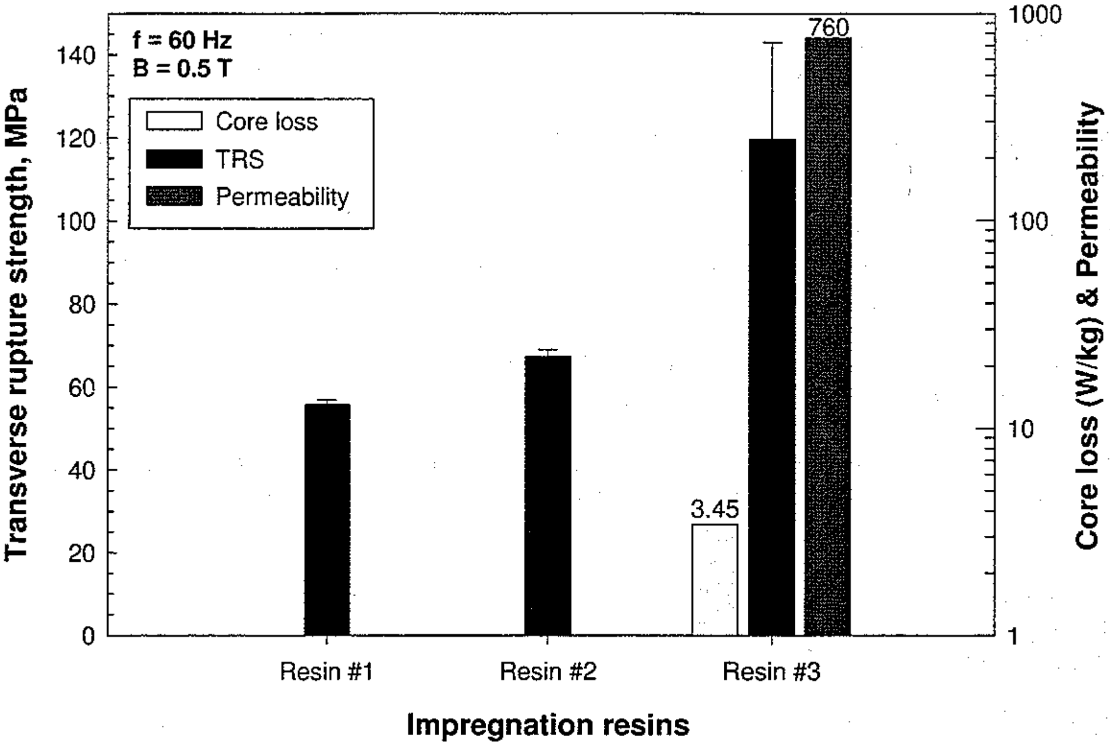


Figure 7: Effect of different impregnation resins on strength and magnetic properties (60 Hz, 0.5 T) of rectangular bars and rings pressed at 620 MPa (45 tsi) and 65 °C and cured in air following the procedure recommended by the resin manufacturers.

4. CONCLUSIONS

This study shows that resin impregnation of high purity iron compacts warm pressed at 65 or 120°C significantly increases their mechanical strength (TRS) without affecting their AC magnetic properties (apparent core loss and permeability). These iron/resin composites can then be used in many AC applications at low frequency (60 Hz). The following conclusions can be drawn from this work:

1- Iron powder compacts impregnated with a phenolic resin and cured 1 hour at 175°C in air give TRS values in the range of 90 to 100 MPa (13,000-14,500 psi), a high apparent permeability (~750) and low core loss 3.5 W/kg (1.6 W/lb);

2- The impregnation parameters (temperature and pressure) evaluated in this study do not significantly affect the TRS, the electrical resistivity and the AC magnetic properties of samples pressed at 65°C or 120°C;

3- The resin composition has a great impact on the mechanical strength of impregnated iron-resin composites.

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