

IRON COMPACTS FOR LOW FREQUENCY AC MAGNETIC APPLICATIONS: EFFECT OF THERMAL TREATMENTS

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ABSTRACT

Parts with good mechanical and AC magnetic properties at 60 Hz can be obtained by pressing a high purity iron powder admixed with a lubricant and heating the parts at moderate temperature ($T < 550^{\circ}\text{C}$) in order to eliminate the lubricant and create bonds between the iron particles. The mechanical strength, electrical resistivity and magnetic properties of the parts are dependent on the interparticle bonds which are governed by the thermal treatment conditions.

The effect of different thermal treatment conditions on the electrical, mechanical and magnetic properties of heat-treated and resin-impregnated specimens prepared with iron-0.75 wt% EBS mixes was studied. Rings and bars were pressed at 620 MPa and submitted to different thermal treatments where atmosphere (N_2 , air and H_2) and temperature ($450\text{-}550^{\circ}\text{C}$) were varied. The best properties were achieved with the specimens treated in N_2 at 500°C .

1. INTRODUCTION

The idea of using insulated iron powders (dielectromagnetics) for the fabrication of magnetic components intended for AC magnetic applications was first proposed by Fritts at the end of the last century. Since then, dielectromagnetics have been used in many different applications [1,2,3,4,5]. Recently, M. Persson and P. Jansson [6] reviewed the principal advantages and limitations of this type of materials for applications in electrical machines. Using dielectromagnetics, components with isotropic properties can be fabricated to near net shape. Dielectromagnetics also have a high electrical resistivity and small eddy-current domains compared to laminations which provide good magnetic properties at moderate and high frequencies. In fact, dielectromagnetics tend to perform better (lower core losses and more stable permeability) than steel laminations at frequencies higher than 100 Hz [6,7,8]. However, for applications at lower frequency, a higher permeability and lower core losses would increase the competitiveness of the materials with lamination assemblies.

Components fabricated from pure iron powder have higher magnetic permeabilities than components made from insulated powders [9]. In fact, the electric insulator generally used in such composites acts as a distributed air-gap in the material [10]. Since the magnetic permeability is strongly influenced by the air-gap thickness, the apparent permeability is greatly reduced when the dielectric content increases. The non magnetic content of the material must then be kept as low as possible to obtain high magnetic permeabilities.

On the other hand, the electrical resistivity of the material must be sufficient to maintain low eddy-current losses when the component is exposed to alternating magnetic fields. Recent works [11] have showed that at low frequency (60 Hz), the minimum electrical resistivity required to minimize eddy-current losses in iron powder compacts is between 1 and 10 $\mu\Omega\cdot\text{m}$. It was also verified that the electrical resistivity of green iron powder compacts is sufficient to maintain low eddy-current losses at 60 Hz [9]. In that case, the insulation is provided by thin air-gaps created after the pressure release at the end of the compaction cycle [12].

Admixed lubricants are generally used for the production of P/M components. The lubricant facilitates the compaction of the powder and allows an easy ejection of the parts after compaction. However, components fabricated with an admixed lubricant generally exhibit low green strength (TRS <15 MPa or 2,000 psi typically) and can not be used in the green state. However, a recent study [13] showed that it is possible to increase the mechanical strength of specimens containing an admixed lubricant using thermal treatment at moderate temperature (30 min at 500°C in N₂). The thermal treatment allows to burnout the lubricant and to create bonds between the iron particles. Using appropriate thermal treatment conditions, it is possible to maintain adequate electrical resistivities for magnetic applications at low frequencies. For applications requiring a higher mechanical strength, the specimens may be resin-impregnated [14].

In this study, an iron powder admixed with 0.75 wt% EBS has been pressed and heated under different conditions. The effect of the atmosphere (N₂, air and H₂) and temperature (450 - 500°C) on the physical, electric, mechanical and magnetic properties of the compacts is evaluated and discussed.

2. EXPERIMENTAL PROCEDURE

A -30/+200 mesh high-purity water-atomized iron powder supplied by QMP was used in the experiments. The powder was dry blended with 0.75 wt% EBS (Ethylene Bisstearamide) in a V-type blender for 30 minutes. Rectangular bars (3.175 x 1.27 x 0.635 cm) and rings (5.26 cm OD, 4.34 cm ID, 0.635 cm thick) were pressed at 620 MPa (45 tsi) in a floating die at 65°C. After compaction, two thirds of the specimens were heated for 30 minutes in different atmospheres (N₂, air and H₂) and at different temperatures (450, 500 and 550°C) to burn out the admixed lubricant and to increase the mechanical strength. Half of the heat-treated specimens were resin-impregnated under vacuum with an epoxy resin to further increase their mechanical properties. After impregnation, the compacts were cured in air at 70°C for 2 hours in order to crosslink the resin. Three bars and two rings were prepared for each experimental condition.

Density, transverse rupture strength (TRS) and electrical resistivity were measured on TRS bars. Density was calculated from the weight (± 0.0001 g) and physical dimensions (± 2.5 μm) of the bars. Transverse rupture 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 (UltraOptec, PM450). Side and thickness effects were taken into account in the resistivity calculations. Five readings were taken on the top and bottom faces of each TRS bar and averaged. DC and AC magnetic properties were evaluated on heat-treated and resin-impregnated rings using an ACT/SMT-500 computer-automated magnetic hysteresisgraph¹. The rings were wound with 534 primary

¹ KJS Associated

turns of #24 gauge copper wire and 143 secondary turns of #30 gauge copper wire for DC characterization and 250 primary turns of #24 gauge copper wire and 250 secondary turns of #30 gauge copper wire for AC magnetic characterization. DC maximum permeability and coercive force were measured for an applied field of 11.9 kA/m (150 Oe) while maximum permeability at 60 Hz and core losses at 60 Hz/1 T were evaluated in AC.

3. RESULTS AND DISCUSSION

3.1 Green properties

The green properties of iron/0.75% EBS bars compacted at 65°C/620 MPa are presented in Table I. Green density of 7.21 g/cm³ and green strength of 21 MPa were obtained. The strength of the green compact is not sufficient for many applications and additional treatments are necessary to increase their mechanical strength.

The electrical resistivity of the specimens is more than two orders of magnitude higher than that of wrought iron: 63 μΩ-m versus 0.1 μΩ-m respectively. The green resistivity is also significantly higher than that of pure iron powder specimens pressed using die wall lubrication ($\rho < 10 \mu\Omega\text{-m}$ [9]). In the present case, the lubricant reduces the interparticle cold welding that normally occurs during compaction and acts as an electric insulator between the iron particles. It is important to mention that the electrical resistivity of the green compact is sufficiently high to minimize the eddy-currents for applications at 60 Hz [11].

Table I: Green properties of iron/0.75% EBS bars compacted at 65°C/620 MPa (no die wall lubrication).

Density	7.21 g/cm ³
Resistivity	63 μΩ-m
TRS	21 MPa (3100 psi)

3.2 Effect of thermal treatment atmosphere

Density

Figure 1 shows the effect of a thermal treatment at 500°C for 30 minutes in different atmospheres on the density and weight loss. The type of atmosphere has no significant effect on the density of the heat-treated specimens. The density of the heat-treated specimens is 0.03 g/cm³ lower than green specimen density. The reduction of the density is mainly due to lubricant burnout as reflected by the weight loss occurring during thermal treatment (Figure 1b).

Substantial differences in weight loss were observed between specimens heated in N₂, air and H₂. For the specimens heated in H₂, the weight loss corresponds to the lubricant content in the mix (0.75 wt% EBS). This observation suggests that the lubricant removal was practically completed after the thermal treatment. The weight loss is slightly lower for the specimens heated in N₂. The smaller weight loss is probably due to the fact that the specimens were slightly oxidized during the thermal treatment in N₂. The small amount of oxygen contained in the gas and the presence of lubricant decomposition products probably favored the surface oxidation of the iron particles during the thermal treatment. This phenomenon was not observed for the specimens heated in H₂ because iron oxides were likely reduced in accordance with the free energies of formation of iron oxides in H₂ at 500°C [15]. For

the specimens heated in air, the weight loss is even smaller because the oxidation was obviously more significant.

Changes to the specimen surface color also suggests that oxidation took place during the thermal treatment in N₂ and air. In fact, the specimens heated in H₂ were bright and clean while those heated in N₂ were slightly darker and those heated in air were dark blue.

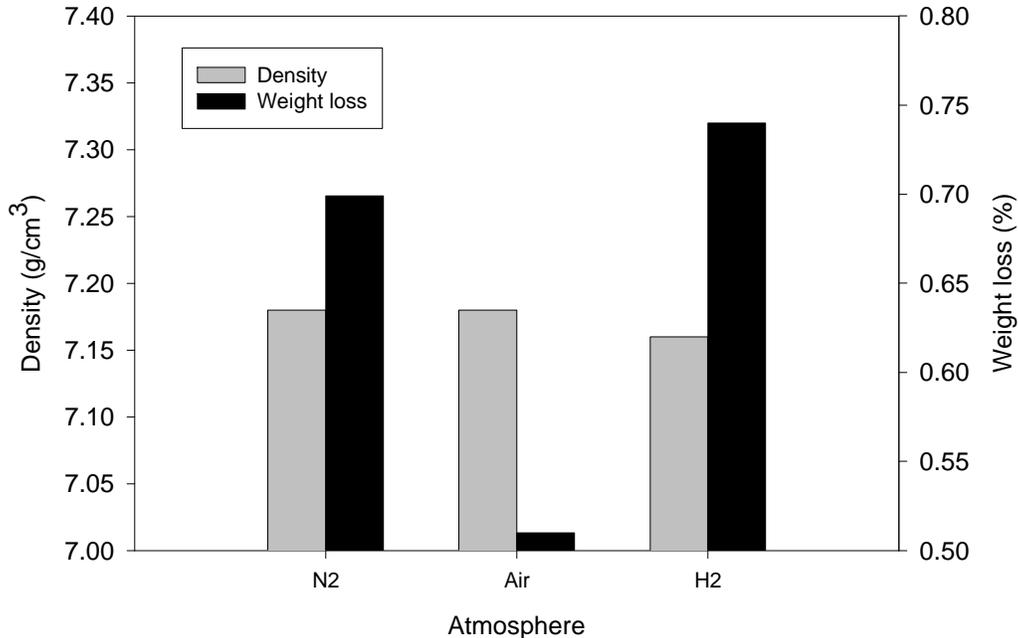


Figure 1. Effect of the thermal treatment atmosphere (30 min at 500°C) on the density and weight loss of iron/0.75% EBS bars compacted at 620 MPa/65°C.

Electrical resistivity

The effect of the thermal treatment atmosphere on the electrical resistivity of the specimens heated at 500°C for 30 minutes is presented in Figure 2. The resistivity of the specimens drastically decreased during the thermal treatment from 63 $\mu\Omega\cdot\text{m}$ in the green state down to less than 1.8 $\mu\Omega\cdot\text{m}$ after the thermal treatment. For the specimens heated in N₂ and air, the electrical resistivity drop may be associated to the formation of interparticle oxide-bonds. Indeed, since the electrical resistivity of iron oxides is lower than that of air, the formation of oxide bonds between the iron particles leads to a reduction of the specimen resistivity when they fill the space previously occupied by air [12]. For the specimens heated in air, this effect is even more important and the resulting resistivity is lower. For the specimens heated in H₂, the very low electrical resistivity is due to a different phenomenon since practically no oxidation took place in this case. The oxide reduction taking place during the thermal treatment likely allowed the formation of metallic contacts between the iron particles (beginning of sintering) thus reducing the resistivity.

Some specimens were resin-impregnated after the thermal treatment to increase their mechanical strength. After impregnation, the electrical resistivity remains unchanged for all specimens. This phenomenon is due to the fact that the resin does not affect the electrical contacts which are created during compaction and the thermal treatment.

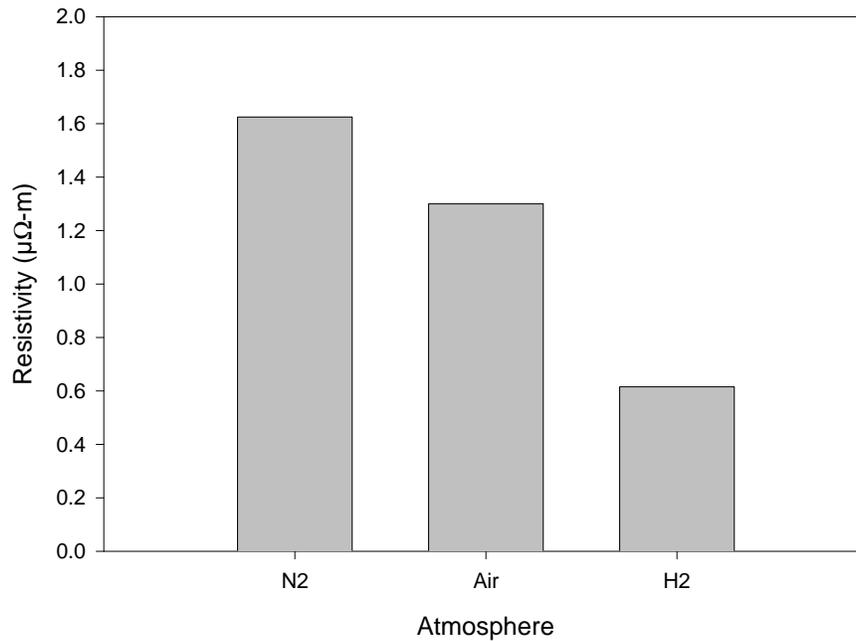


Figure 2. Effect of the thermal treatment atmosphere (30 min at 500°C) on the electrical resistivity of iron/0.75% EBS bars compacted at 620 MPa/65°C.

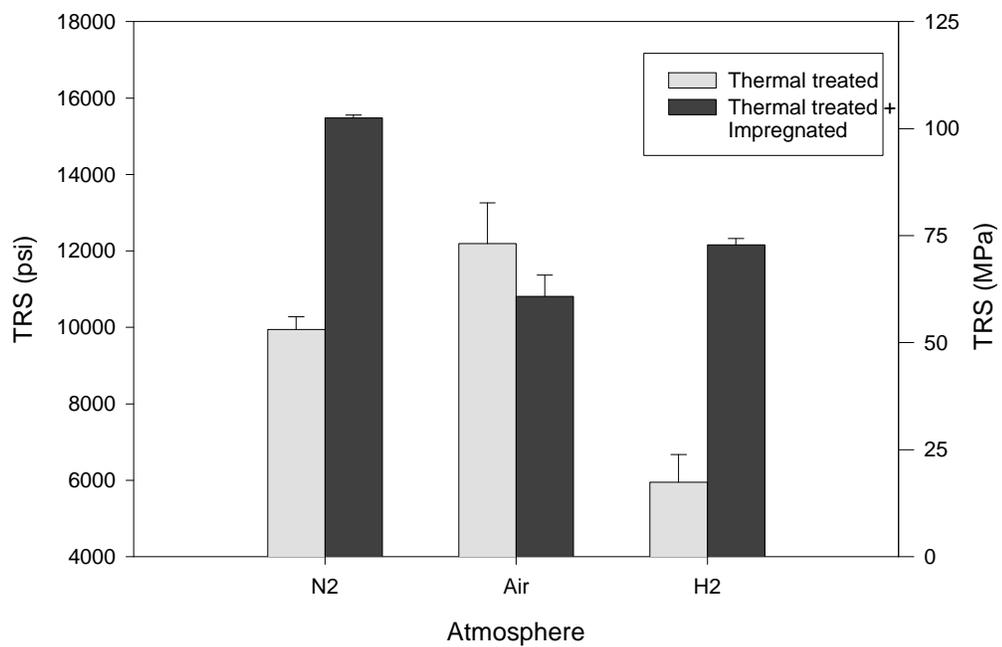


Figure 3. Effect of the thermal treatment atmosphere (30 min at 500°C) on the strength before and after resin-impregnation (iron/0.75% EBS bars compacted at 620 MPa/65°C).

Mechanical strength

The effect of the thermal treatment atmosphere on the transverse rupture strength of the specimens is shown in Figure 3. The strength of the heat-treated specimens is in all cases higher than the green

strength of the material (21 MPa/3100 psi). The strength increases up to 70 MPa (10,000 psi) for the specimens heated in N₂ and up to 80 MPa (12,000 psi) for the specimens heated in air. This increase in strength is due to the oxide bonds formed between the iron particles during the thermal treatment. For specimens heated in H₂, the increase in strength is less important and a value of 40 MPa (6,000 psi) was obtained. These results suggest that, for the conditions used, the oxide bonds formed in air and N₂ atmospheres are stronger than the metallic bonds created during thermal treatment in H₂.

Figure 3 also shows the effect of resin-impregnation on the mechanical strength of heat-treated specimens. Resin impregnation increased the strength of the specimens heated in N₂ and H₂, up to 110 MPa (16,000 psi) and 80 MPa (12,000 psi) respectively. In these cases, the resin flowed between the iron particles during impregnation and contributed to particle bonding after curing. This bonding provides an increase of strength by approximately 40 MPa (6,000 psi). Surprisingly, the resin-impregnation did not improve the strength of the specimens heated in air. The oxidation of the specimens treated in air is more important and the oxides partially closed the pores and impeded the infiltration of the resin between the particles. The smaller weight increase during the resin impregnation of the specimens heated in air (0.03 wt% vs ~1 % for the specimens heated in N₂) supports that assumption.

Magnetic properties

Figure 4 shows the effect of the thermal treatment atmosphere on the DC magnetic properties at an applied field of 11.9 kA/m (150 Oe) for heat-treated specimens after resin-impregnation. Similar DC maximum permeabilities were obtained for the specimens heated in N₂ and air (about 440) but lower permeability was obtained for the specimen heated in H₂ (380). This difference in DC maximum permeability is difficult to explain. It could be related to the thickness of the distributed air-gap or to the nature of the interparticle contacts. Additional works are needed to elucidate the mechanisms responsible of such variations.

The coercive force H_c is not significantly affected by the type of atmosphere. The stability of H_c with the atmosphere is due to the fact that the coercive force is more dependent on the composition and structure of the magnetic material (carbon content, residual stress, grain size) than on the thickness of the air-gap [16,17]. Moreover, at 500°C, the type of atmosphere does not strongly affect the composition and structure of the iron powder as much as it does at higher temperature [18].

The AC magnetic properties at 60 Hz of iron/0.75% EBS specimens heat-treated and resin-impregnated are presented in Figure 5. The properties of the specimen heated H₂ is not presented since its electrical resistivity was not sufficiently high for characterisation at 60 Hz. The AC maximum permeability and core losses (at 60 Hz) of the specimens heated in N₂ and air are consistent with the results obtained in DC characterization. The maximum permeability of the specimen heated in air is slightly higher than that of the specimen heated in N₂: 695 versus 680. Core losses at 1 T are similar for the specimens heated in air and N₂ with values around 11 W/kg.

It is worth mentioning that the maximum permeability obtained with the heat-treated specimens is significantly higher than that of untreated iron-resin dielectromagnetics (typically < 300) [19]. The high permeability of the present materials is attributed to the lower thickness of the distributed air-gap in the material and to a stress relief which take place during thermal treatment. It is known that the stresses induced in the specimens during compaction decrease the permeability and increase the coercive force [20]. The thermal treatment at 500°C likely permits partial relief of these stresses thus increasing the permeability and decreasing the coercive force. Actually, the coercive forces reported in Figure 4 are significantly lower than that of untreated iron/resin dielectromagnetics [19]: 300 A/m versus 400 A/m.

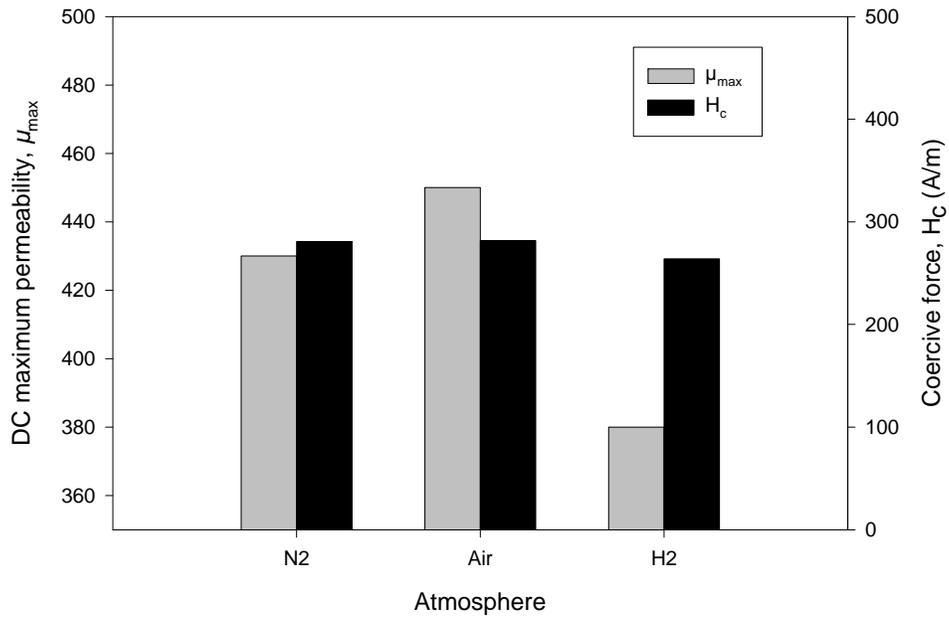


Figure 4. Effect of the thermal treatment atmosphere on the DC maximum permeability and coercive force ($B = 11.9$ kA/m or 150 Oe) for rings pressed at 620 MPa/65°C, heated at 500°C for 30 min and resin-impregnated.

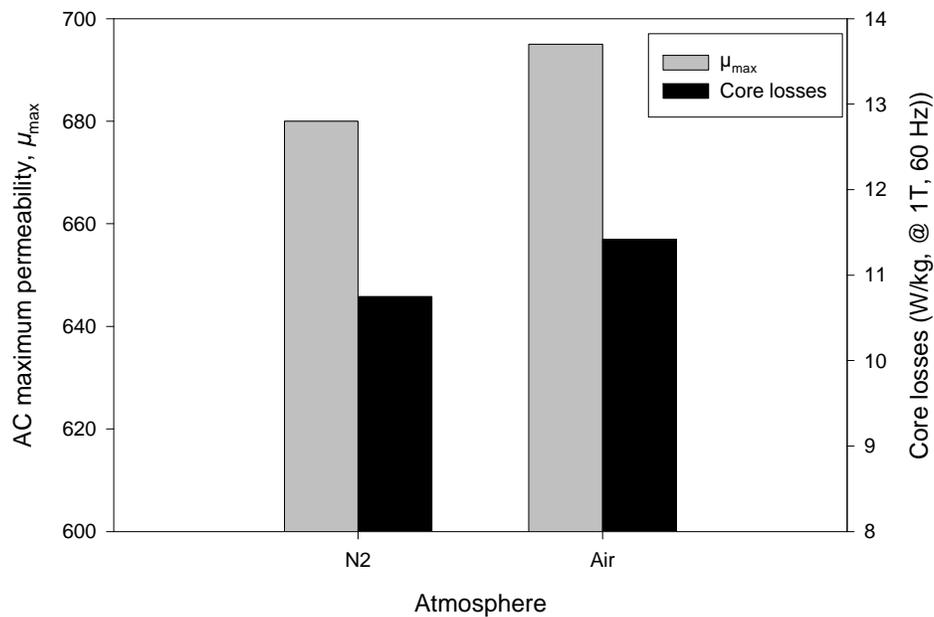


Figure 5. Effect of the thermal treatment atmosphere on the AC maximum permeability at 60 Hz and core losses (at 60 Hz/1 T) for rings pressed at 620 MPa/65°C, heated 30 min at 500°C and resin-impregnated.

3.3 Effect of thermal treatment temperature

Density

Figure 6 shows the effect of the thermal treatment temperature (450, 500 and 550°C) on the density and weight loss after a treatment in N₂ for 30 min. The results indicate that temperature has very little effect on the density and weight loss of the specimens in the narrow temperature range studied. As observed in the preceding section, the net weight loss (~ 0.70 wt%) is the result of two phenomena: a weight loss due to the lubricant burnout and a weight gain due to iron powder oxidation.

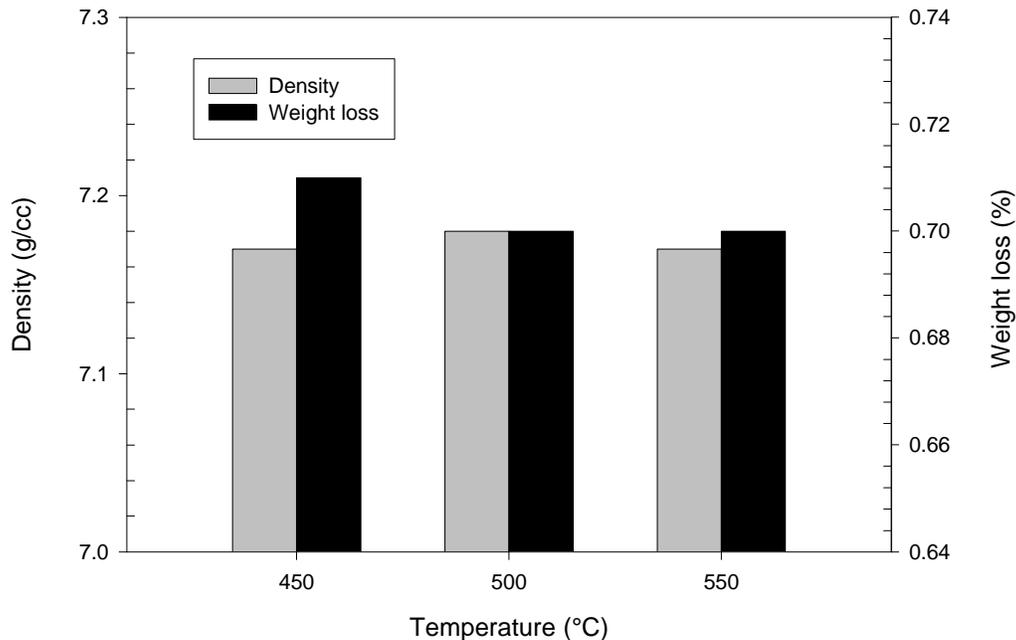


Figure 6. Effect of the thermal treatment temperature (30 min in N₂) on the density and weight loss of TRS bars compacted at 620 MPa/65°C.

Electrical resistivity

The effect of the thermal treatment temperature on the electrical resistivity of specimens heated in N₂ for 30 minutes is shown in Figure 7. The electrical resistivity decreases from 3.0 μΩ-m down to 1.2 μΩ-m as the thermal treatment temperature increases from 450°C up to 550°C. This decrease in resistivity is probably due to a modification in the quality or composition of the interparticle oxide bonds and to the creation of metal-to-metal contacts.

Mechanical strength

Figure 8 presents the effect of thermal treatment temperature and resin-impregnation on the transverse rupture strength of iron/0.75% EBS bars heated in N₂ for 30 minutes. As previously mentioned, the thermal treatment allows a significant increase in the strength of the specimens from 20 MPa (3,000 psi) in their green state, up to about 70 MPa (10,000 psi) after thermal treatment. According to results of Figure 8, the strength increases slightly with temperature: from 55 MPa (8,000 psi) at 450°C up to 70 MPa (10,000 psi) at 550°C. As shown in the previous section, the resin-impregnation also has a very positive effect and virtually doubles the mechanical strength of the specimens to reach values higher than 16,000 psi/110 MPa.

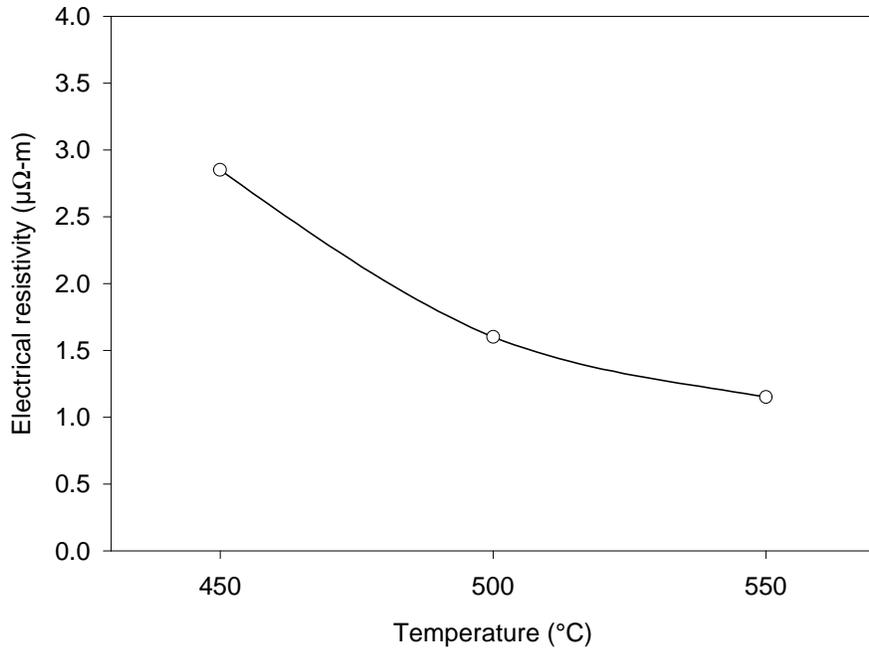


Figure 7. Effect of the thermal treatment temperature (30 min in N₂) on the electrical resistivity of bars compacted at 620 MPa/65°C.

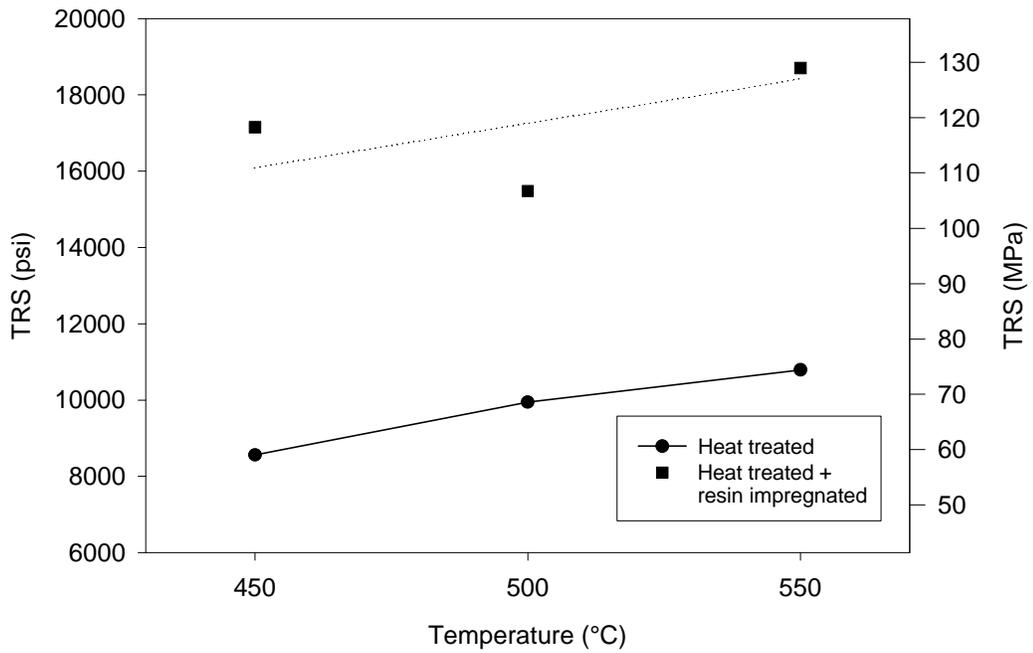


Figure 8. Effect of the thermal treatment temperature (30 min in N₂) on the strength before and after resin-impregnation of bars compacted at 620 MPa/65°C.

Magnetic properties

The DC magnetic properties of the specimens heated at different temperatures in N₂ for 30 minutes are given in Figure 9. The maximum permeability increases from 400 up to 470 and the coercive force decreases from 320 A/m down to 250 A/m when the thermal treatment temperature increases from 450°C up to 550°C. As previously discussed, these improvements in permeability and coercive force are related to a stress relief during thermal treatment.

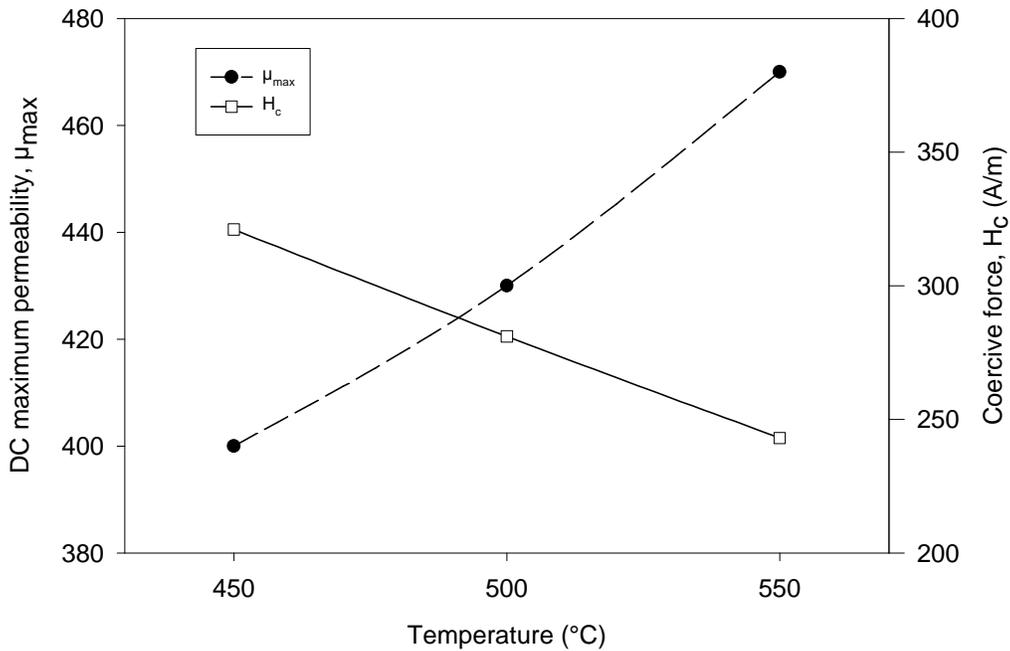


Figure 9. Effect of the thermal treatment temperature (30 min in N₂) on maximum permeability and coercive force H_c ($B = 11.9$ kA/m or 150 Oe) of rings compacted at 620 MPa/65°C.

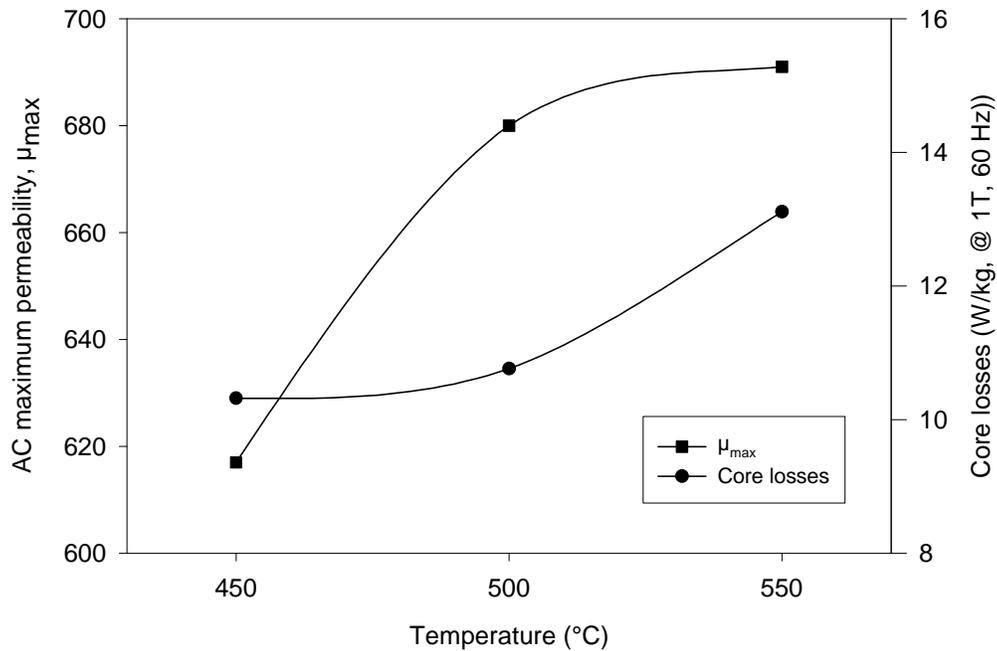


Figure 10. Effect of the thermal treatment temperature (30 min in N₂) on the AC maximum permeability at 60 Hz and core losses (1 T/60 Hz) of rings compacted at 65°C/620 MPa.

The AC magnetic properties at 60 Hz of the specimens heated at different temperatures in N₂ for 30 minutes are presented in Figure 10. The AC maximum permeability increases with the thermal

treatment temperature but seems to attain a plateau at 550°C with a value of 690. This saturation in permeability may come from the increasing amount of eddy-currents when the heat-treatment temperature increases. Indeed, when temperature increases, the electrical resistivity decreases (Figure 7) and this gives rise to eddy-currents which reduce the apparent permeability. The effect of the increasing amount of eddy-currents may also be observed on the core losses in Figure 10. The losses increase from 10.2 W/kg at 450°C up to 13.3 W/kg at 550°C. When the specimens are heated 30 minutes at 550°C in N₂, the resistivity is too low to minimize the eddy current losses at 60 Hz.

4. CONCLUSION

Pure water-atomized iron powders admixed with a lubricant can be used to fabricate soft magnetic components having interesting mechanical and magnetic properties at 60 Hz. The powder can be shaped into soft magnetic components using a simple technology like uniaxial compaction under standard conditions. Treating the components at moderate temperature ensures a burn out of the lubricant and increases the mechanical strength of the material. In addition, resin-impregnation after the thermal treatment allows a further increase in the mechanical strength of the material.

The study evaluated the effect of the heat treating conditions on the properties of iron compacts intended for low frequency applications (60 Hz). The results indicated that the temperature (in the 450°C to 550°C range) and type of atmosphere (N₂, H₂ and air) of the thermal treatments significantly affected the properties of the material. The results led to the following observations and conclusions:

- The electrical resistivity of the specimens heated in H₂ (30 min at 500°C) is too low for magnetic applications at 60 Hz.
- The specimens heated in air have good mechanical and magnetic properties but can not be resin-impregnated to further increase their mechanical strength.
- The best properties were achieved with the specimens heated in N₂. After a thermal treatment at 500°C in N₂ for 30 minutes, a magnetic permeability of 680 (60Hz), core losses of 11 W/kg (60 Hz/1 T) and mechanical strength of 70 MPa (10,000 psi) was obtained. When the specimens were resin-impregnated, the mechanical strength was higher than 110 MPa (16,000 psi).

5. ACKNOWLEDGEMENTS

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