IRON COMPACTS FOR LOW FREQUENCY AC MAGNETIC APPLICATIONS: EFFECT OF LUBRICANTS

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ABSTRACT

The use of high-purity iron powder without any additive allows the production of parts with high density and electrical resistivity suitable for AC magnetic applications at low frequency (60 Hz). Components with good soft magnetic properties can also be fabricated using iron powder admixed with lubricants. The mixes are easy to shape and do not require die wall lubrication. A thermal treatment at moderate temperature (30 min at 500°C in N_2) burns out the lubricant and increases the strength while retaining adequate electrical resistivities for low-frequency soft-magnetic applications.

This paper describes the effect of different lubricants on properties of pressed iron powder intended for low-frequency AC magnetic applications (60 Hz). The electrical, mechanical and magnetic properties measured on green, thermal-treated and resin-impregnated specimens are presented. The results indicate that the nature of the lubricant significantly affects the properties of the materials.

1. INTRODUCTION

The feasibility of using powder metallurgy (P/M) for the production of AC soft magnetic components has long been known [1,2,3,4,5]. Recent developments in P/M have renewed the interest in soft magnetic P/M components intended for alternating current (AC) applications and composites materials (dielectromagnetics) are now commercially available for those applications [6]. These materials have isotropic electrical and magnetic properties. The isotropic magnetic properties offer the possibility of controlling the spatial distribution of the magnetic flux in the component. These materials have high electrical resistivities and small eddy-current domains which provide good magnetic properties at moderate and high frequencies. In fact, dielectromagnetics tend to perform better than steel laminations at frequencies higher than 100 Hz [7,8,9]. However, for low frequency AC soft magnetic applications, the materials must have high permeability, low core losses and acceptable mechanical strength to compete with steel laminated assemblies.

The selection of appropriate materials (iron powder, insulation, lubricant) and processing conditions (shaping technology, compaction temperature and pressure, thermal treatment) allow the production of components with tailored properties suitable for a wide range of applications. The choice of the material and manufacturing process is usually based on the material's performance and production costs.

High-purity water-atomized iron powder has good compressibility and is an adequate material for the fabrication of AC soft magnetic components. Previous works have shown that the electrical resistivity of green iron powder compacts is sufficient to maintain low eddy current losses at 60 Hz [10]. For that reason, iron particles do not necessarily have to be coated with an electrical insulator for applications at 60 Hz. When an insulating coating is applied on iron powder for AC magnetic applications, it acts as a distributed air-gap between the magnetic particles and significantly reduces the permeability of the material [11]. Indeed, the maximum permeability of green compacts fabricated with a pure iron powder is higher than the permeability of parts fabricated with an insulated iron powder [12]. In addition, coating the powder with an insulator represents an additional operation that generally increases the cost of the final components.

To take full advantage of P/M production techniques, lubrication problems encountered in the fabrication of dielectromagnetics should be eliminated [13,14]. These problems may be solved by using die wall lubrication systems. However, such techniques are not applicable for all types of part geometry and they generally reduce the production rate. The use of admixed lubricants facilitate the compaction and ejection of the parts but generally reduces the green strength.

Previous works have shown that a thermal treatment at low temperature in the presence of oxygen strengthens green iron compacts fabricated with die wall lubrication while maintaining adequate electrical resistivities for AC soft magnetic applications at low frequencies [15]. Preliminary studies showed that such thermal treatments can be done on specimens pressed from iron powder admixed with a lubricant. The thermal treatment temperature is chosen to allow the decomposition of the lubricant and to permit the formation of interparticle bonds that increase the mechanical strength while retaining adequate insulation for AC soft magnetic applications. When higher mechanical strength is required, a resin-impregnation treatment can also be done [16].

This paper presents the effect of different lubricants on the properties of specimens compacted from iron-0.75 wt% lubricant mixes. The specimens were characterized after pressing in their green state, after a thermal treatment at 500°C in N_2 and after resin-impregnation. The effect of the lubricants on the electrical, mechanical and magnetic properties (DC and 60 Hz) is presented and discussed.

2. EXPERIMENTAL PROCEDURE

A -30/+200 mesh high-purity water-atomized iron powder supplied by QMP was used in these experiments. The powder was dry blended with four different lubricants (0.75 wt%) in a V-type blender for 30 minutes. A description of the lubricants used is given in Table I.

Table I: Description of the different lubricants used.

Lubricant	Description	Supplier
EBS	Ethylene Bisstearamide Acrawax C, Atomized	Lonza inc.
PEG	Polyethylene Glycol Carbowax 8000	Union Carbide Co.
AlSt	Aluminum Stearate	Witco Co.
ZnSt	Zinc Stearate	Witco Co.

Rectangular bars (3.175 x 1.27 x 0.635 cm) and rings (OD = 5.26 cm, ID = 4.34 cm, t = 0.635 cm) were pressed at 620 MPa (45 tsi) in a double action floating die at 65°C. After compaction, two thirds of the specimens were heated at 500°C for 30 min in N_2 to burn out the admixed lubricant and to increase the mechanical strength of the compacts. Half of the heated specimens were resin-impregnated under vacuum with an epoxy resin to further increase their mechanical strength. After impregnation, these compacts were cured in air at 70°C for 2h in order to cross link the resin. Three bars and two rings were prepared for each experimental condition.

Density, transverse rupture strength (TRS) and electrical resistivity were measured on the TRS bars. The density was calculated from the weight (± 0.0001 g) and the 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 (UltraOptec PM450) adapted for this application. Five readings were taken on the top and bottom faces of each TRS bar and averaged. Side and thickness effects were taken into account in the resistivity calculations.

DC and AC magnetic properties were evaluated using an ACT/SMT-500 computer-automated magnetic hysteresisgraph[1]. Rings were wound with 534 primary 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. Maximum permeability and coercive force for an applied field of 11.9 kA/m (150 Oe) were measured in DC while maximum permeability (60 Hz) and core losses (60 Hz/1 T) were measured in AC.

3. RESULTS AND DISCUSSION

Density

Figure 1 shows that the green density of the compacts is affected by the type of lubricant used. The highest green density is obtained with PEG and the lowest with ZnSt. The density drop recorded during the thermal treatment is related to the lubricant burn out. For instance, the top surface of as-pressed and thermal-treated specimens fabricated with EBS is shown in Figure 2. On the green specimen, EBS can be seen on the surface while after thermal treatment, the lubricant is no longer visible into the pores. The weight loss presented in Figure 1b gives an indication of the extent of lubricant removal during the thermal treatment. The weight loss is higher for the specimens containing synthetic waxes (EBS and PEG) than for specimens containing stearates (AlSt and ZnSt). The differences may come from incomplete lubricant removal. In fact, the specimens containing EBS were clean and bright after the thermal treatment while those fabricated with the stearates were covered with apparent residues. This observation suggests that the delubrication of the specimens fabricated with EBS was completed while residues remained in the specimens fabricated with the stearates. It is well known that EBS burns out cleanly while ZnSt leaves decomposition products such as ZnO that cannot be eliminated at low temperature [17]. Figure 3 shows the fracture surface of thermal-treated specimens fabricated with the different lubricants. Small spots are visible on the fracture surface of the specimens containing the stearates.

The weight losses measured after the thermal treatment are, in all cases, smaller than the lubricant content of the compacts (0.75 wt%). This difference may be attributed to specimen oxidation during the thermal treatment. In fact, the fracture surface of the thermal-treated specimens are all darker than the fracture

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surface of the green compacts, suggesting that surface oxidation of particles took place during the thermal treatment. This effect was previously observed in specimens fabricated with pure iron-powder compacts heated in air at 175°C [15]. The oxidation may come from residual oxygen in the gas which was used or from lubricant decomposition products.

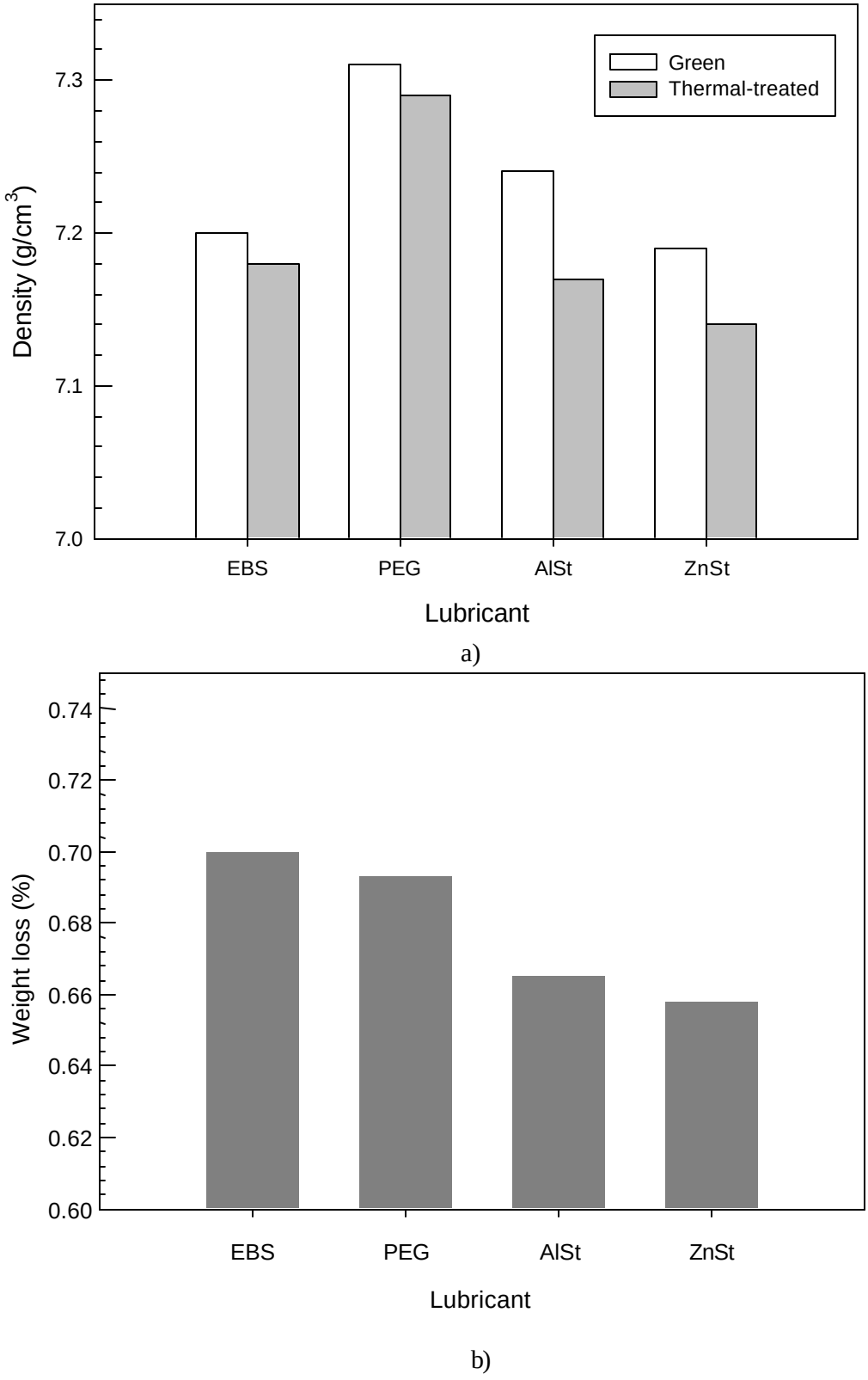
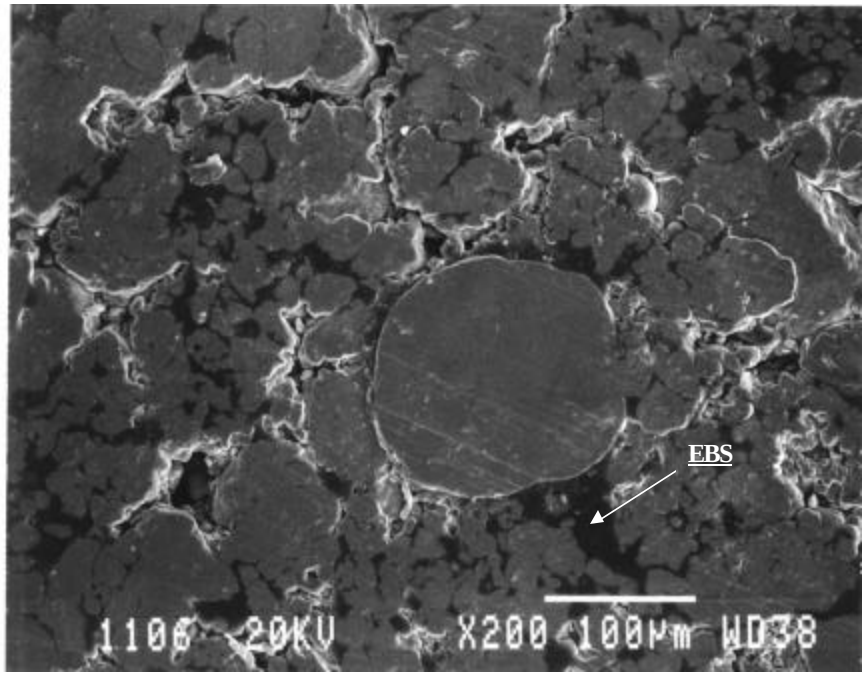
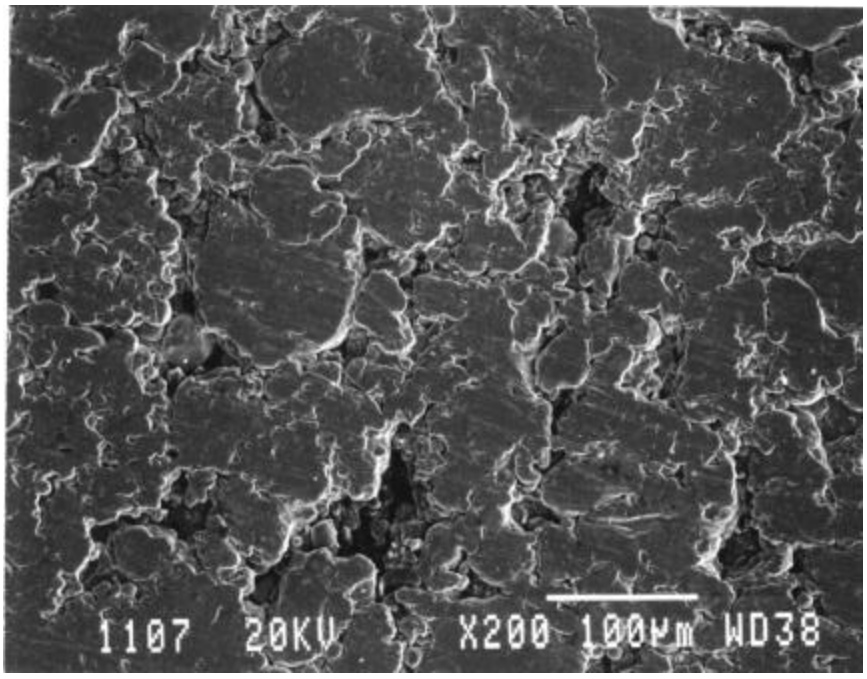


Figure 1. Effect of the type of lubricant on a) the density and b) the weight loss during the thermal treatment at 500°C for 30 minutes in N_2.



a)



b)

Figure 2. Top surface of specimens fabricated with 0.75 % EBS pressed at 620 MPa/65°C: a) as-pressed and b) after a thermal treatment at 500°C in N_2 for 30 minutes.

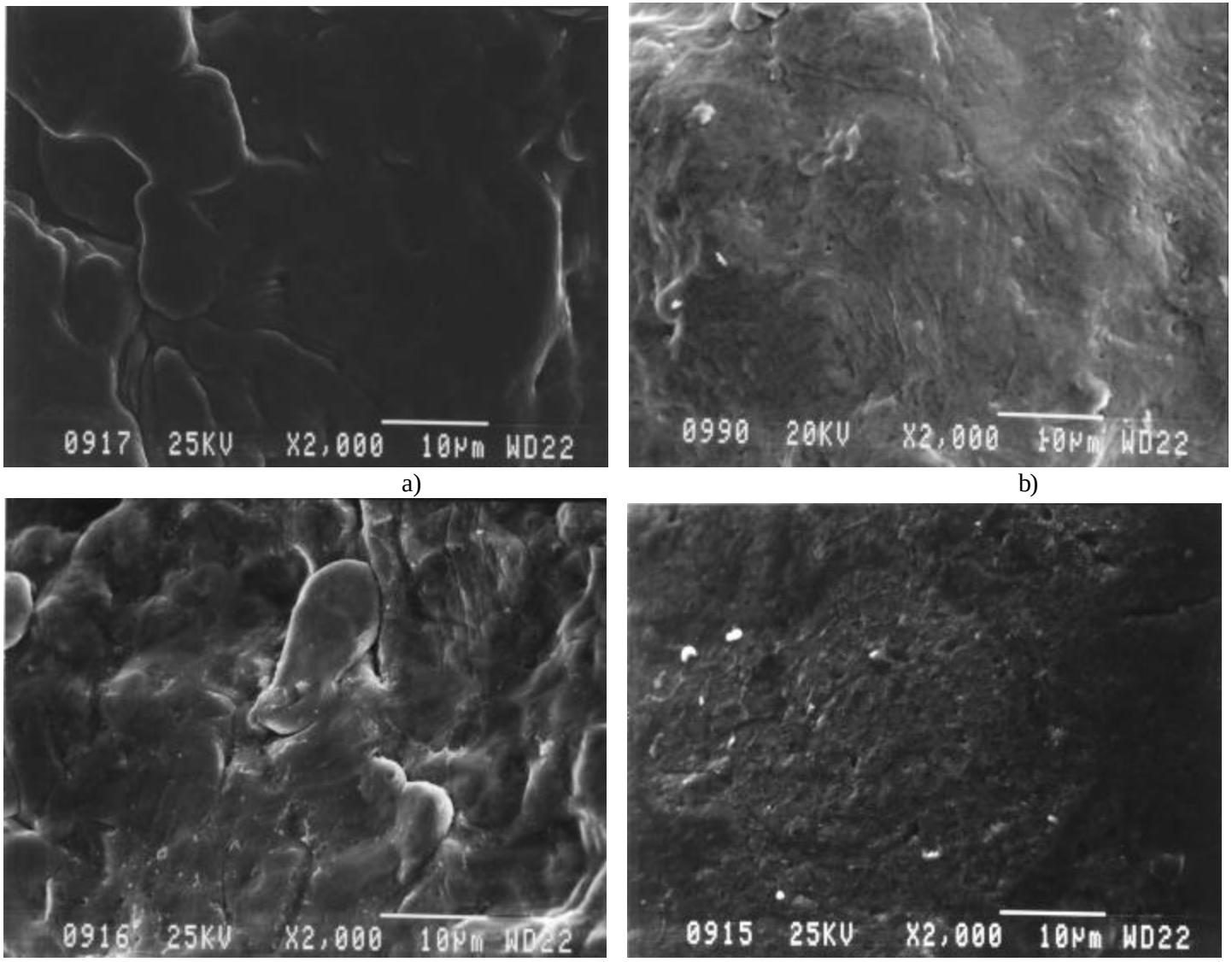


Figure 3. Fracture surface of TRS bars fabricated with different lubricants (pressed at 620 MPa/65°C and heated 30 minutes at 500°C in N_2): a) EBS b) PEG c) Zn-St d) Al-St.

Electrical resistivity

Figure 4 shows the effect of the type of lubricant on the electrical resistivity of specimens as-pressed, after thermal-treatment and after resin-impregnation. The resistivity of the green specimens is much higher than that of pure iron-powder compacts fabricated under similar conditions using die wall lubrication ($\rho < 10$ μΩ-m typically [10]). The high electrical resistivity of the green specimens is related to the presence of gaps between the iron particles. In fact, the lubricant reduces the formation of interparticle electrical contacts during compaction and acts as an insulating layer in the green compacts. As shown in Figure 4, electrical resistivity of the green compacts depends on the type of lubricant used. For example, the electrical resistivity of specimens fabricated with ZnSt (~100 μΩ-m) is about four times higher than that of specimens fabricated with PEG (~25 μΩ-m). These differences are likely related to differences in density and to the lubricant distribution within the compacts.

Figure 4 shows that the electrical resistivity significantly drops after thermal treatment. This drop may be associated to the formation of oxide bonds between the iron particles during thermal treatment. In fact, previous works [15] showed that interparticle oxide bonds are formed when iron powder compacts are heated in the presence of oxygen. In the present case, the oxygen may come from the presence of impurities in the N_2 gas and from the lubricant decomposition products. Indeed, since the resistivity of iron oxides is lower than that of air, the formation of oxide bonds leads to a reduction of the specimen resistivity when the oxide fills the space previously occupied by air. After thermal-treatment, the specimens fabricated with the different lubricants do not have the same electrical resistivity. The highest electrical resistivity was measured on specimens fabricated with ZnSt (4 μΩ-m). The high resistivity of the specimens containing ZnSt is associated to their lower density and to the composition of the interparticle interfaces which probably contains resistive ZnO. It is worth noting that the resistivity of the thermal-treated specimens fabricated with EBS and ZnSt is much higher than that of wrought iron (~ 0.1 μΩ-m) and may be sufficient for many AC soft magnetic applications at low frequency. The effect of the electrical resistivity on the magnetic properties of the materials will be developed in more detail later in this paper.

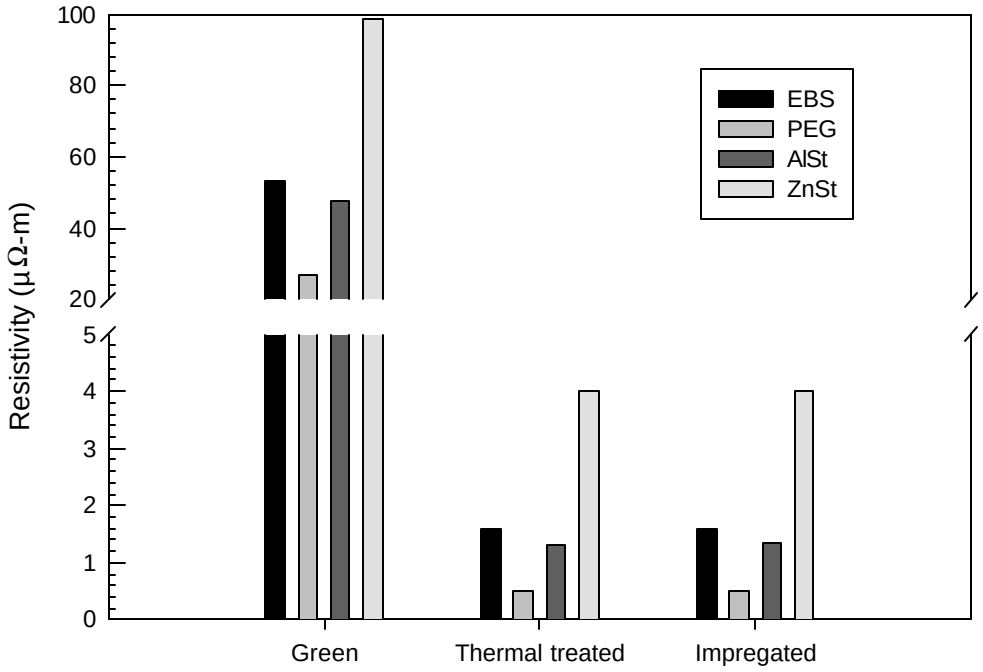


Figure 4. Effect of a thermal-treatment at 500°C for 30 minutes in N_2 and resin-impregnation on the electrical resistivity of specimens pressed at 620 MPa/65°C from mixes containing 0.75 % of different lubricants.

Figure 4 shows that the impregnation has little effect on the electrical resistivity of the compacts. This phenomenon was previously observed for pure iron compacts fabricated using die-wall lubrication [16]. The electrical resistivity of the compacts is more dependent on the amount and the quality of the electrical contacts formed during compaction and thermal treatment than by the electrical resistivity of the insulator itself (air vs resin).

Mechanical strength

The transverse rupture strength of green, thermal-treated and resin-impregnated specimens fabricated with the different lubricants is presented in Figure 5. As previously reported by other authors [18, 19], the nature of the lubricant affects the green strength. The highest green strength is obtained with the specimens containing PEG: 40 MPa (6,000 psi) vs 20 MPa (3,000 psi) for EBS. The higher green strength of the specimens fabricated with PEG may be attributed to their higher density (Figure 1) and to the highest degree of interparticle cold welding during compaction as suggests the electrical resistivity measurements (Figure 4).

Figure 5 shows that the strength significantly increases after the thermal treatment at 500°C. In fact, a strength of about 70 MPa (10,000 psi) was obtained with the specimens fabricated with admixed EBS and PEG. The increase in strength confirms that some bonds were created during thermal treatment. As previously mentioned, these bonds are formed by thermal oxidation during the thermal treatment. This phenomenon was previously observed by T.Werber [20] who showed that interparticle oxide bonds formed during a thermal-treatment at moderate temperature leads to cohesive bonds between iron particles. The lowest strength of the specimens fabricated with the metallic stearates may be attributed to the lower density of the specimens and to the presence of residues which impedes the formation of strong interparticle bonds during thermal treatment.

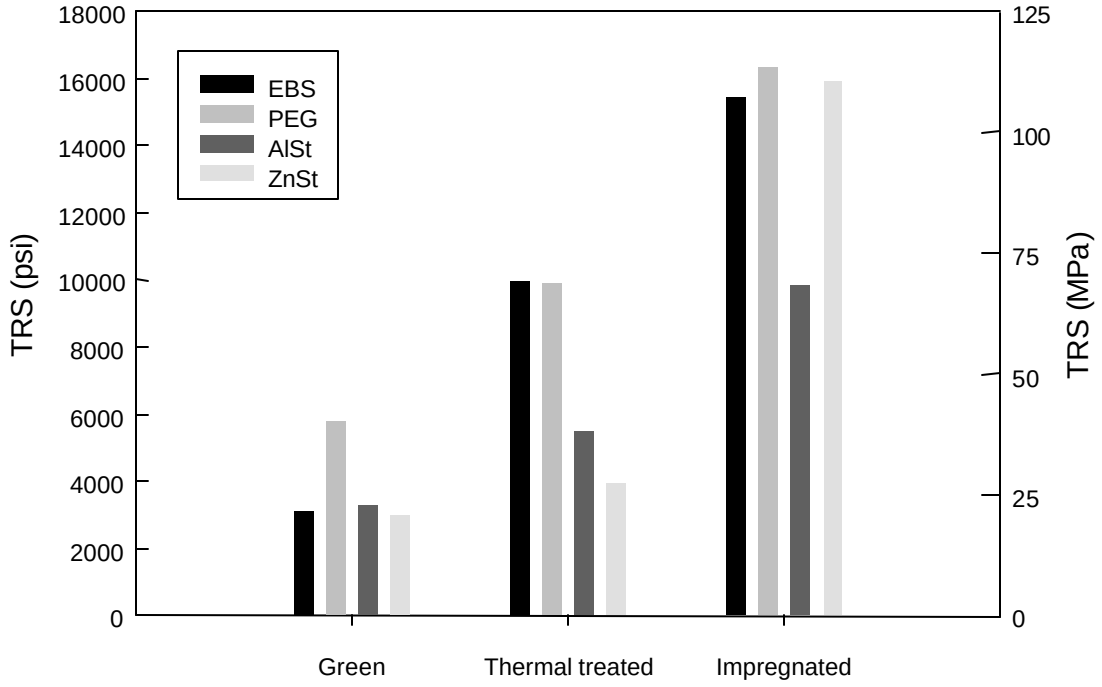
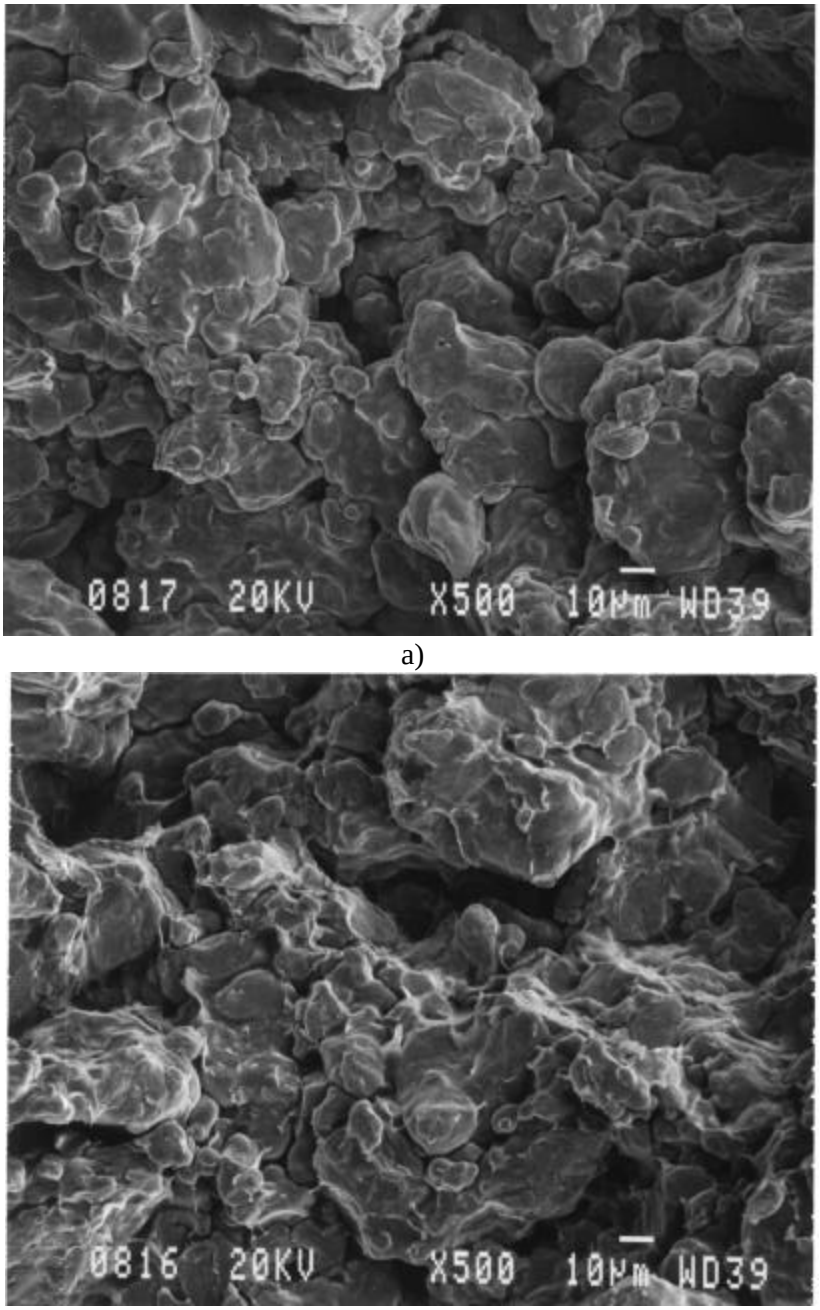


Figure 5. Transverse rupture strength of specimens compacted at 620 MPa/65°C using iron-0.75 % lubricant mixes.

A resin-impregnation of the thermal-treated specimens further increases the strength of the specimens (Figure 5). Values up to 110 MPa (16,000 psi) were obtained with all the specimens except for those containing AlSt. The lower strength of the specimens fabricated with AlSt was attributed to the presence of residues in the compacts which impede the resin impregnation and prevent the formation of good iron-resin bonds. In fact, the weight variation measured during resin-impregnation was smaller for the specimens containing AlSt (0.3 % vs 0.9 % for EBS). For the specimens containing EBS, PEG and ZnSt, the resin flows between the iron particles within the parts as shown in Figure 6 for a specimen fabricated with EBS. The porosity network left by the lubricant after the thermal-treatment is filled by the resin during impregnation. After curing at 70°C, the resin acts as a binder and increases the strength of the specimens.



a)

b)

Figure 6. Micrographs taken at the center of the fracture surface of iron-0.75 % EBS specimens pressed at 620 MPa/65°C and heated at 500°C for 30 minutes in N_2: a) after the thermal treatment and b) after resin impregnation.

Magnetic properties

Figure 7 shows that the DC maximum permeability depends on the type of lubricant used. Permeability varies from 340 for the specimens pressed from iron-ZnSt mixes up to 500 for iron-PEG mixes. The maximum permeability is strongly dependent on the thickness of the distributed gap in the material (or sample density), which is in turn related to the type of lubricant used. The maximum permeability is also strongly dependent on the internal stresses in the material. In fact, it is well known that stresses induced during compaction decrease the permeability and increase the coercive force of the material [21]. Thus, if the material is stress relieved, the permeability increases and the coercive force decreases. This phenomenon explains why the thermal-treated materials have relatively high permeability and low coercive

force as compared to untreated dielectromagnetics. In fact, coercive forces around 280 A/m are obtained for all the thermal-treated specimens while values around 430 A/m were measured on untreated composites fabricated under similar conditions with the same iron powder [22]. Figure 7 also shows that the coercive force is not strongly affected by the type of lubricant used. Unlike permeability, the coercive force is not dependent on the air-gap thickness or the density [23, 24] but rather on the composition and structure of the magnetic material (residual stress, grain size).

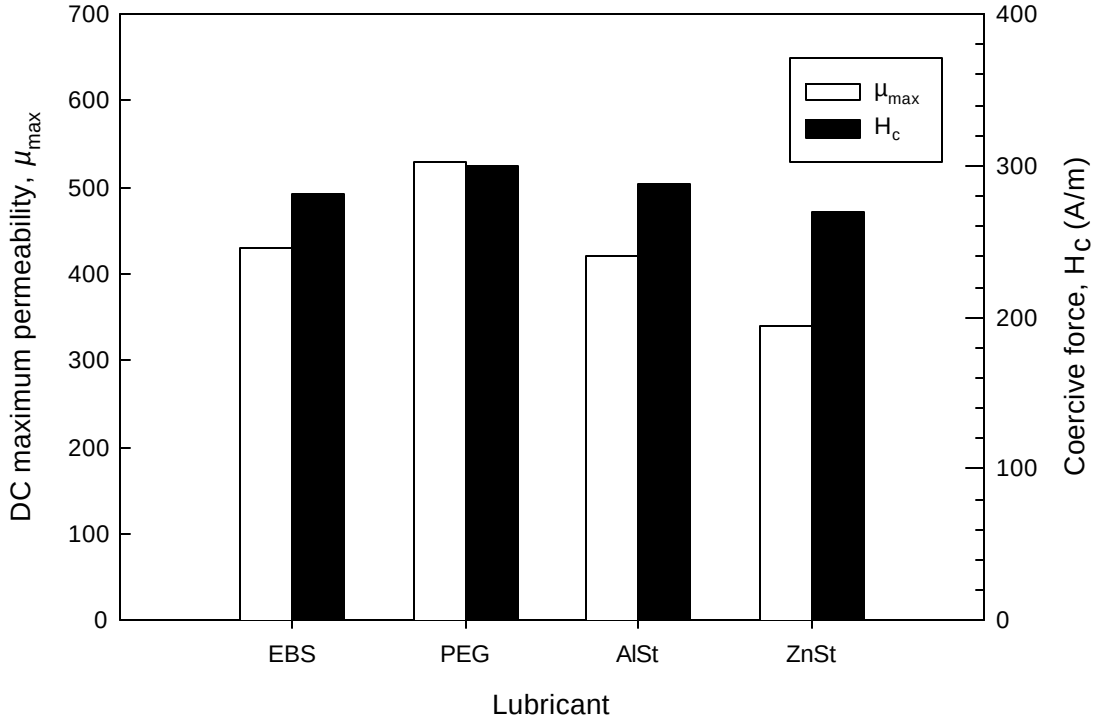


Figure 7. DC maximum permeability and coercive force H_c (B = 11.9 kA/m or 150 Oe) of specimens pressed at 620 MPa/65°C, heated 30 min at 500°C in N_2 and resin-impregnated.

The AC permeability (60 Hz) of the specimens depends on the type of lubricant used, as shown in Figure 8. The maximum permeability varies from 520 for the specimens pressed from iron-ZnSt mixes up to 730 for iron-PEG mixes. As mentioned previously, the variation is mostly due to the variation of the thickness of the distributed air-gap in the material. The thickness of the distributed air-gap depends on the other hand on the density of the specimens (Figure 1) as well as the presence or distribution of lubricant residues in the specimens. Actually, the presence of non-magnetic residual products (like ZnO) between iron particles should negatively affect the material permeability.

As shown in Figure 8, the core losses are affected by the type of lubricant used. In fact, core losses varying from 10 to 11 W/kg at 60 Hz/1 T were measured on the specimens fabricated with EBS, AlSt and ZnSt while core losses around 17 W/kg were measured on the specimens fabricated with PEG. These variations come from the eddy-currents, which are inversely proportional to the electrical resistivity of the materials. When the electrical resistivity is not sufficient to maintain low eddy-currents, as for the specimens fabricated with PEG, the core losses increase.

It is worth noting that the core losses at 60 Hz measured on the specimens fabricated with EBS and St-Zn are similar to what is generally measured with iron-resin composites fabricated under similar conditions [22]. However, for applications at higher frequencies, the electrical resistivity of the thermal-treated specimens will not be sufficient to maintain low eddy-current losses and insulated iron powder should be used.

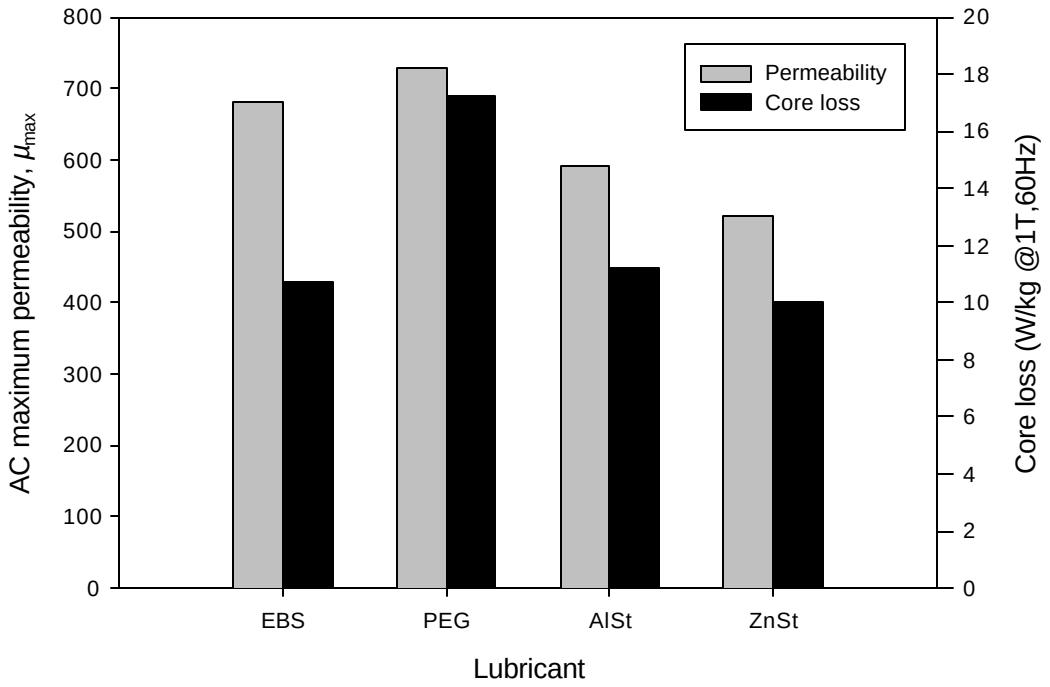


Figure 8. AC permeability (60 Hz) and core losses (1 T/60 Hz) of specimens pressed at 620 MPa/65°C, heated 30 min at 500°C in N_2 and resin-impregnated.

4. CONCLUSION

It is possible to fabricate soft magnetic components having interesting mechanical and magnetic properties using pure water-atomized iron powder admixed with a lubricant. After compaction, the specimens are heated to burn out the lubricant and to increase their mechanical strength. After thermal-treatment, the specimens may be resin-impregnated to further increase their mechanical strength.

The effect of different lubricants on the properties of iron compacts intended for low frequency applications (typically 60 Hz) was evaluated. The results indicated that the nature of the lubricant significantly affected the properties of the materials. The results led to the following observations and conclusions:

- For 60 Hz applications, the specimens fabricated with PEG were not sufficiently resistive after thermal treatment at 500°C for 30 min in N_2.

- Following thermal treatment at 500°C for 30 min in N_2, the specimens fabricated with AlSt contained lubricant residues which impeded the formation of strong interparticle bonds and limited resin-impregnation.

- Specimens fabricated with EBS had high maximum permeability (680 at 60Hz), low core losses, 11 W/kg (60 Hz/1 T) and acceptable mechanical strength (110 MPa after resin-impregnation).

- Specimens fabricated with ZnSt had maximum permeability of around 520 at 60Hz, low core losses, 10 W/kg (60 Hz/1 T) and acceptable mechanical strength (110 MPa after resin-impregnation). The specimens containing ZnSt had higher electrical resistivity than the specimens fabricated with the other lubricants. Thus, ZnSt is an interesting lubricant for applications where eddy-current losses must be minimized.

5. ACKNOWLEDGEMENTS

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