

Study of the Electromagnetic Properties of a Soft Magnetic Composite (SMC) composed of iron particle with thin layer of phosphate coating and its efficiency when applied to a low frequency magnetic field

This work presents the study of the preparation routes of a soft magnetic composite (SMC), its electromagnetic properties and efficiency when applied to induction motors (low frequency magnetic fields), as an alternative to traditional electric steels. Thus, different SMCs (such as pure iron powder and ASC 100.29 iron powder, both with coating and isolation of the particles with phosphoric acid) have been studied in comparison with commercialized SMCs, such as Somaloy 700 3P. In addition, methodologies for depositing the insulating layer, such as physical deposition, spraying and the Sol-Gel process were also studied. Initially, a commercial powder sample and another sample with a thin layer of phosphate were prepared. Sintered parts of these SMCs were then prepared and, subsequently, their electromagnetic parameters were acquired. From these data, an analysis of the magnetic flux in the engines built by these materials was made, in which it was observed that the sample of the composite material had properties compatible with conventional steels. It was also observed that, with the improvement of the SMC preparation routes, they can be used in the development of new engine topologies, for the most diverse applications, without loss of efficiency, mainly with regard to low frequency magnetic applications.

Keywords: Soft Magnetic Composite, Coated Iron Powder, Electrical Machines, Insulating Thin-Layers.

1. Introduction

In recent years, soft ferromagnetic composites, also known as (SMC) are being widely studied due to the many advantages they offer over electromagnetic steel sheets in relation to their isotropic magnetic properties, high electrical resistivity, design flexible, potential for size reduction and high design flexibility [1]. To use powdered magnetic cores in AC (Alternating Current) magnetic field applications, however, it is important to reduce the loss of iron, that is, the sum of eddy current loss and hysteresis loss.

Magnetic powder cores are therefore manufactured by compacting magnetic powder particles and coating with insulating layers to prevent the formation of eddy currents. This coating technique with an insulating structure between the particles is crucial to improve the magnetic properties of an AC motor core. More modern technologies and processes allow the properties of these materials to be improved to provide advanced magnetic properties, high permeability and saturation magnetization, but with high electrical resistivity in order to minimize the losses due to eddy currents [2].

* Corresponding author: Jaime A. Back, Master of Electrical Engineer – Federal University of Rio Grande do Sul, PortoAlegre, Brazil, E-mail: jaime.back@gmail.com

¹ Electrical Engineer; PPGE3M Department, Federal University of Rio Grande do Sul – UFRGS, Porto Alegre, Brazil

² Mechanical Engineer; Prof. Dr. Ing. PPGE3M Department, Federal University of Rio Grande do Sul – UFRGS, Porto Alegre, Brazil

³ Mechanical Engineer; PPGE3M Department, Federal University of Rio Grande do Sul – UFRGS, Porto Alegre

The concept of SMC is based on the coating of magnetic particles with a surface layer, on the order of nanometers, electrically insulating and subsequently joined in a three-dimensional matrix form of a finished compact. In this way, 3D magnetic properties are acquired due to the isotropic nature of this material, thus offering freedom in creating project concepts (design flexibility), in unique and innovative applications [3 - 5].

The isotropic nature of the SMC combined with the possibilities of conformation allows the planning of parts with unusual and three-dimensional geometries, offering advantages over traditional laminated electric steels [2]. Thus, alloys of soft magnetic materials, composed of iron powders, together with other elements, such as phosphorus, silicon or nickel, can be used in solid core engines, constructed from laminated steel sheets [6, 7].

In this context, magnetic particles, such as cobalt, iron, ferrites such as Fe_3O_4 and $\gamma\text{-Fe}_2\text{O}_3$, among other oxides, have shown potential applications in several fields, including iron-fluids, industrial applications, robotics, environmental remediation, biomedical and diagnostic and therapy areas, such as nuclear magnetic resonance, among other applications [8, 9]. Laminated silicon steels have a high permeability, but their losses are increased due to the increase in frequency. Soft Ferrite has a low magnetic loss at high frequencies, but its low permeability requires the use of a lot of material.

The manufacturing process of these electric machines is, therefore, more expensive compared to the casting process, for example, a process that would result in massive cores [10], since new perspectives indicate that soft magnetic material alloys, obtained from of base iron powder, mixed with other elements, may present properties superior to those obtained from laminated steel sheets. The soft magnetic materials, produced by Powder Metallurgy (M/P), are the target of different studies and applications, in electronics and mainly in electric motors, due to the large portion of electrical energy that they consume [6, 10 - 14].

2. Material and Methods

This chapter deals with a brief review of the fundamentals of magnetism, the classification of magnetic materials with an emphasis on ferromagnetic materials. Losses in electromagnetic applications are also discussed. An introduction to the powder metallurgy process is made, as well as the SMCs, highlighting its properties, advantages and disadvantages in relation to other soft magnetic materials and their applications.

2.1. Soft Magnetic Composites

Manufactured by powder metallurgy (M/P), soft magnetic composite materials are materials with electromagnetic properties and that have two or more elements in their composition. These materials are generated from tiny particles of iron powder covered by a dielectric material, which produces electrical insulation to the grains (Figure 1.b). The atomized iron powder is mixed with lubricating material, placed in a matrix and then compacted to generate “green” pieces of ferromagnetic material, as shown in the sequence in Figure 1.c.

After this process, the molded body obtained by compaction is heat treated at a temperature of at least 200°C and not higher than the thermal decomposition temperature of the insulating film covering the particle. In the case of an insulating film based on phosphoric

acid, for example, the thermal decomposition temperature of the insulating film is 500°C. This heat treatment is carried out in order to reduce the distortions formed inside the molded body during the compaction operation [14, 18].

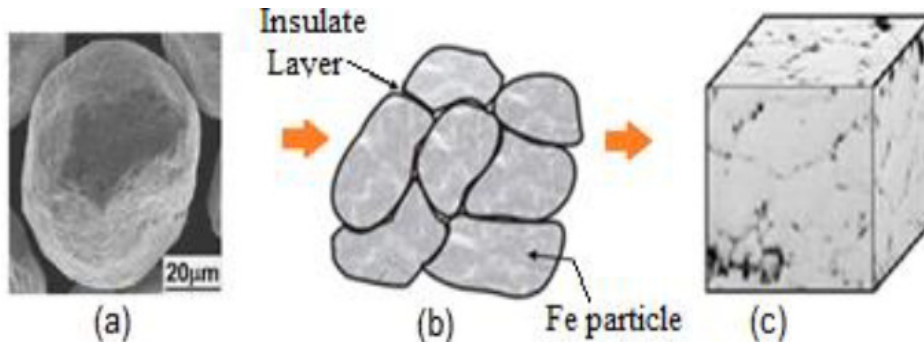


Figure 1: Illustration of the SMC creation process [18]

The insulating coating that separately separates the iron powder particles in an SMC product is the fundamental feature of this technology. Its thickness, coverage and strength under the different processing operations are fundamental aspects for the properties of a magnetic part. This insulating coating has the main purpose of increasing the resistivity and/or electrical performance between the particles of the ferromagnetic material used in the (SMC). In most cases, as ferromagnetic material (SMC particle core), pure iron, or low-alloyed iron powder, with varying particle size distribution, produced using traditional water atomization techniques, is used.

The illustration in Figure 2 shows a particle of atomized magnetic iron type Somaloy® covered by dense nano-layers for isolation between particles using a unique Sol-Gel process method [19].

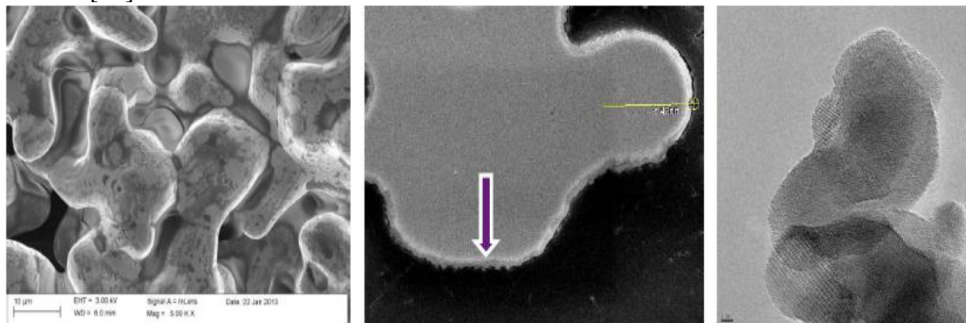


Figure 2: Illustration of a magnetic atomized iron particle covered by an insulating nano-layer [19]

2.2. Magnetic Losses of SMC

Ferromagnetic materials are those that, when subjected to an external magnetic field, present a resultant magnetization much more intense than the applied field and still maintain some remaining magnetization even after the external field is removed [16, 17].

Eddy currents, also known as eddy currents, are induced in any conductive material by an alternating magnetic flux [17]. Considering an alternating field and a uniform material

(disregarding demagnetization effects, skin effect, among other structural effects) the currents will occur as shown in Figure 3, perpendicularly to the direction of the variant magnetic field.

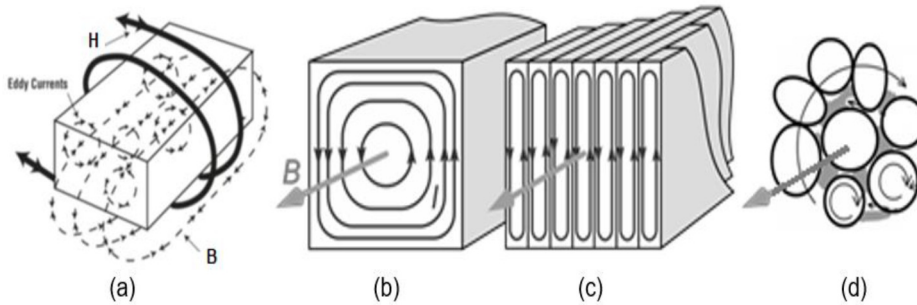


Figure 3: (a) Illustration of how eddy currents are generated in a conductive material. (b) Eddy currents in a massive conductive material. (c) Eddy currents in isolated plates and (d) Eddy currents in a particulate magnetic composite [17]

2.3. Preparation of the SMC insulating layer

The metallic magnetic particle can be formed, for example, of iron (Fe), an alloy based on iron and silicon (Fe Si), alloy based on iron and nitrogen (Fe N), alloy based on iron and nickel (Fe Ni), iron and carbon based alloy (Fe C), iron and boron based alloy (Fe B), an iron and cobalt based alloy (Fe Co), an iron and phosphorus based alloy (Fe P), or a alloy based on iron, aluminum and silicon (Fe Al Si). In addition, the metallic magnetic particle can be an individual metal or an alloy [20, 21].

Some examples of materials that can be used for the organic layer include: a thermoplastic resin, such as a polyimide; a thermoplastic polyamide; a thermoplastic-imide polyamide; polyphenylene sulfide; polyamide-imide; polyethersulfone (PES); polyethylenamide (PEI) or polyester ketone; a non-thermoplastic resin, such as high molecular weight polyethylene; an absolute aromatic polyester or an absolute aromatic polyimide; and higher acid-based materials such as zinc stearate, lithium stearate, calcium stearate, and so forth [17, 18].

A powder mixture is then obtained by mixing the composite magnetic particles and the organic substance. There are no special restrictions on the mixing process. Some examples of procedures that can be used include: vibrating ball mill, planetary ball mill, co-precipitation, chemical vapor deposition (CVD), physical vapor deposition (PVD), electrodeposition, high vacuum spraying, vaporization and Sol-Gel procedure [21].

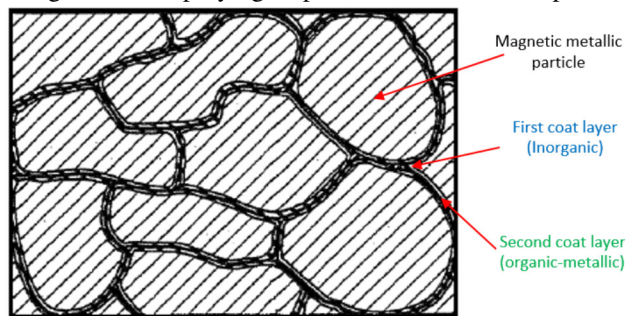


Figure 4: SMC molded by M/P. (a) Illustration of the iron particle being coated. (b) Thin layer of silicon dioxide covering the particle after heat treatment [20]

The SMC shown in figure 4 is composed of:

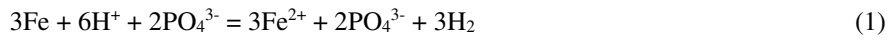
- Core base (Fe particle): Particles of iron soft magnetic core can be derived from a water atomized iron powder, a gas atomized iron powder or an iron sponge powder. However, the use of a water atomized powder is preferred. Iron-based soft magnetic core can be chosen from a set consisting of an essentially pure iron, a Fe-Si alloy iron with up to 7% by weight (preferably up to 3% by weight of silicon), a selected ferroalloy from the sets formed by Fe-Al, Fe-Si-Al, Fe-Ni, Fe-Ni-Co, or their combinations [20]. An essentially pure iron is preferred, and the average particle size should be between 25 and 600 μm , preferably between 45 and 400 μm , more preferably between 60 and 300 μm for a good permeability x resistivity ratio.
- Inorganic layer (first layer of the coating): The core particles are provided with a first inorganic insulating layer, which is preferably based on phosphorus. This first coating layer can be achieved by treating an iron-based powder with phosphoric acid dissolved in water or organic solvents. To a water-based solvent, corrosion inhibiting agents and surfactants are optionally added. The phosphating treatment can be repeated. The inorganic phosphorus-based insulating coating of the iron-based core particles is preferably carried out without any addition such as dopants, corrosion inhibitors or sulphatants [21].
- Organic-metallic layer (second coating layer): At least one organic-metallic layer is located outside the first phosphorus-based layer. The organic-metallic compound has an alkaline character and can also include coupling properties, that is, a so-called coupling agent that will be coupled to the first inorganic layer of the iron-based powder. The substance must neutralize excess acidic acids and by-products of the first layer. If coupling agents chosen from the group consisting of alkoxysilanes, titrates, aluminates or zirconates are used, then the substance will be hydrolyzed and partially polymerized (some of the alkoxy groups will be hydrolyzed with the subsequent formation of alcohol). It is also believed that the coupling or crosslinking properties of organic-metallic compounds will couple them to the metallic or semi-metallic compound in the form of particles, which can improve the mechanical stability of the compacted composite component [20, 21].

2.4. The phosphating process

Phosphating is a well-known metal conversion treatment, especially in the automotive industry. It is used to provide the material with a thin layer of a phosphate coating that gives the surface anti-corrosion and wear resistance properties. In addition, it has been shown that such a coating provides insulation if used in iron powder, significantly improving its properties, composition, homogeneity, among others.

According to Kopeliovich (2013), the main components of a conventional phosphating solution are water or acetone as the main solvent, phosphoric acid (H_3PO_4), ions (typically cations) of divalent metals such as Zn^{2+} , Fe^{2+} , Mn^{2+} and an accelerator (generally is an oxidizing reagent, such as nitrate, nitrite or peroxide).

When a metallic material, for example iron, is immersed in a phosphate solution, iron ions are supplied by the dissolving substrate and a top chemical reaction occurs on the surface. There the dissolution of the iron is initiated in the micro anodes present in the substrate by the free phosphoric acid present in the bath. The evolution of hydrogen occurs at microcathodic sites according to the following equations [22]:



As these reactions proceed, the pH is reduced locally on the surface. Then, the metal surface becomes first covered by a thin layer of primary ferrous phosphate that is soluble in water or acetone, and then a thicker secondary and tertiary insoluble ferrous phosphate given by the reactions below:



Considering eq. (4) as primary ferrous phosphate (soluble), eq. (5) secondary ferrous phosphate (insoluble) and eq. (6) tertiary ferrous phosphate (insoluble).

2.5. Powder metallurgy process

Unlike other metallurgical processes, powder metallurgy does not have a liquid phase or only partially present during the process. It is a manufacturing technique that allows parts to be produced in definitive or practically definitive formats, often without the need for other types of finishing, such as machining. The components obtained from this process have structural and physical characteristics that are impossible to achieve with any other process. The basic processes of (M/P) can be divided into obtaining powders, mixing, compacting and sintering, as shown in the schematic representation of Figure 5. Sometimes complementary steps, such as rectification, are necessary.

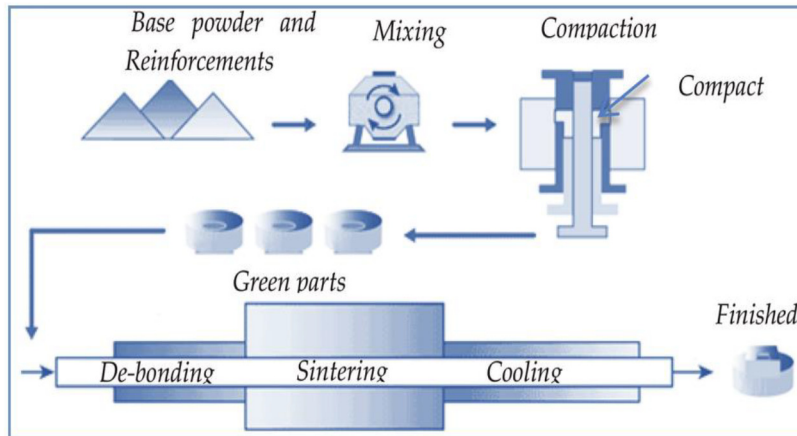


Figure 5: Conventional powder metallurgy process

In M/P, the powders after being mixed are compacted in matrices where they acquire the shape of the matrix cavity. Afterwards, they are placed in sintering furnaces where they acquire consistency and mechanical resistance. It should be noted that powders of different chemical nature can be obtained, as long as they are mixed homogeneously.

3. Problem formulation

The development of a research route, simulation of the magnetic elements raised from the produced materials should assist in a future conception of an electric induction motor (rotor and stator) with new technologies and new design, associated with performance analysis via Finite Element Methods (FEM).

3.1. The Iron Coating Process

Eddy currents loss can be minimized with the insulating coating of each particle providing very small eddy current paths within a particle and a relatively high resistivity of the bulk material. The small non-magnetic distances between each particle act as air gaps and decrease the permeability of the bulk material.

When using the phosphating method to provide insulating coatings, a layer thickness more suitable for electromagnetic applications should be sought. For example, using concentrations of phosphoric acid that provide improved electrical insulation properties, but resistivity can be increased to some extent.



Figure 6: Schematic diagram of the eddy current loss reduced by the effectively integrated insulation coating layer

The amount of phosphoric acid dissolved in the solvent must correspond to the desired coating thickness on the coated powder particles, as defined below. An adequate concentration of phosphoric acid in acetone has been found to be between 30 ml to 100 ml of phosphoric acid per liter of acetone. The appropriate amount of solution (acetone + phosphoric acid) to be added is 50 to 150 ml for 1000 grams of iron powder.

Consequently, it is preferred to use only phosphoric acid in a solvent at such concentrations and treatment times in order to obtain the indicated relationship between the size of the particles, the thickness of the layer and its homogeneity. Afterwards the powder is dried in an oven at 80 °C for 30 minutes.

3.2. Sample analysis

Two samples were prepared by the phosphating method, using the method of immersing the iron powder in a liquid solution of acetone + phosphoric acid. Equal amounts of iron powder were immersed in different concentrations of phosphate, for 5 minutes, then dried in 85 °C for 45 minutes. The SEM micrograph images of the two samples after compaction can be seen below.

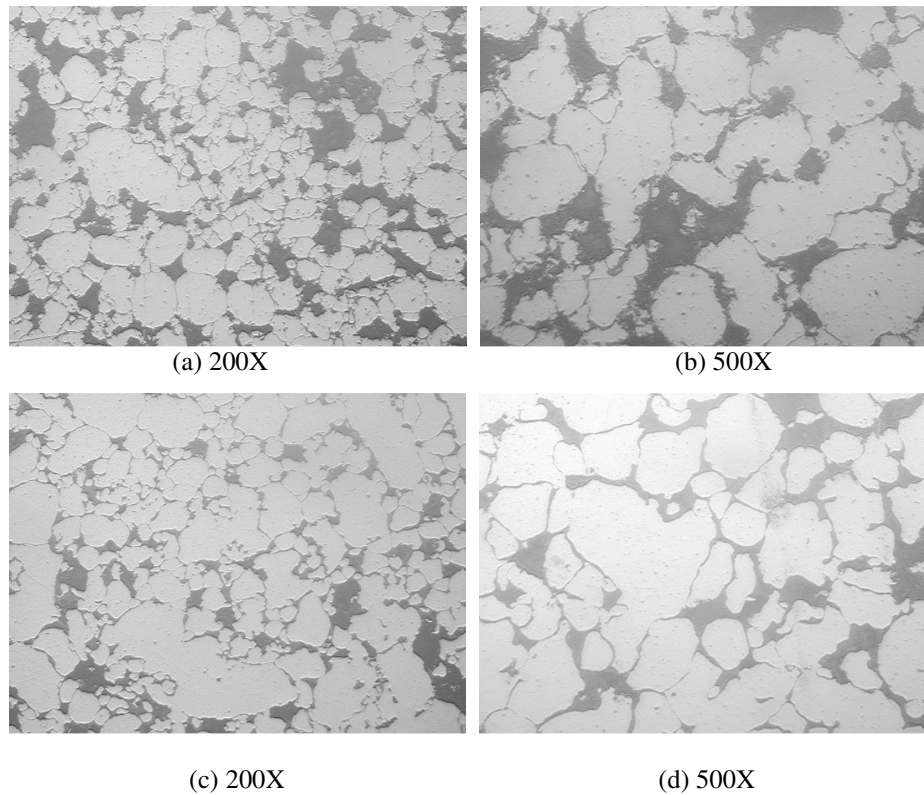


Figure 7: Samples with dosage 1/10 parts of phosphate (a and b). Samples with dosage 1/20 parts of phosphate (c and d)

SEM micrographs of iron powders after the chemical coating process with phosphoric acid are shown in Figure 7.a and Figure 7.b represent samples with a 1/10 mixture of phosphoric acid. Figure 7.c and Figure 7.d represent samples with a 1/20 parts of phosphate mixture.

From the micrographs it was possible to observe the decrease in the thickness of the phosphate layer covering the iron particle. This decrease is important because it increases the magnetic permeability of the SMC, thus increasing its magnetic flux density. However, the material still has greater electrical resistance than the Soma alloy powder.

3.3. Electromagnetic characterization of SMC

The characterization of these materials can be done by means of specimens, which provided the analysis of the physical and magnetic properties of each material, comparing these with existing results in literature [9, 13 - 16].

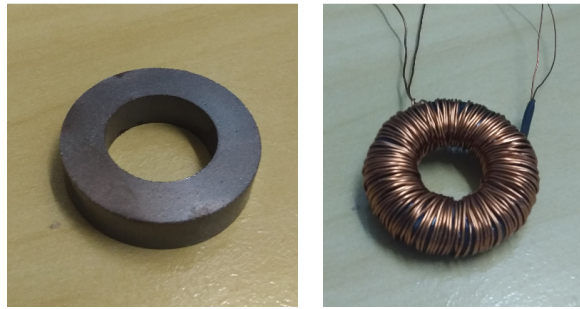


Figure 8: The specimens in the form of a ring

For the hysteresis test, it is necessary to prepare the sample, as shown in Figure 6, and then in the winding (winding of copper wires) of primary and secondary turns in the ring, known as the Roland Ring, according to the standard ASTM A773/A773M-01 [23]. The procedure consists of: insulating the ring with plastic film to prevent stripping of the enameled wire, winding of secondary turns AWG 26 and then new insulation, followed by winding of primary turns AWG 23.

After preparing (coiling) the sample, the test to obtain the hysteresis and magnetization curve is still based on the procedures of the ASTM A773 / A 773M-01 standard, using appropriate equipment and software for data acquisition, model TLMP-FLX-D, from the company Global Mag. During the magnetization curve tests of the material, also known as the DC test, excitation currents of 5A were used with a frequency of 0.05 Hz. In order to obtain the curve and hysteresis, a frequency in the 10 Hz – 100Hz range was used, according to specifications of the manufacturer's manual [23].

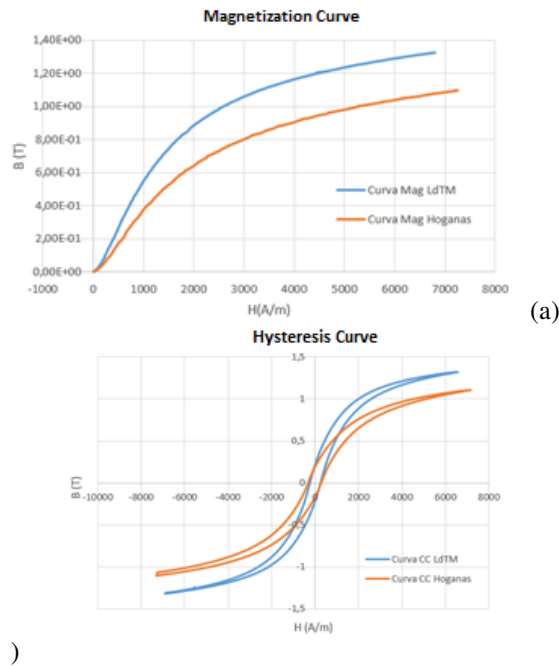


Figure 9: Measurement of the hysteresis curve using the ring method

Figure 9 shows the magnetization curves and hysteresis lessons for the samples produced. The blue line is the commercial material Somaloy 700 3P (Hoganas) and the red line is the phosphatized powder. It is possible to notice a decrease in both B_{max} (maximum magnetic flux density), shown in Figure 9.a, where we have a $B_{max} = 1.38$ Tesla for the Somaloy material and 1.14 Tesla for the phosphatized material.

Regarding hysteresis losses, shown in Figure 9.b, the total region of hysteresis losses remains practically the same at the frequency of 10 Hz, and the phosphatized SMC presents significant increases in losses for frequencies above 50 Hz, due mainly the thickness of the phosphate layer.

3.4. Simulation with FEMM

As proposed in this work, a simulation environment was created to test the sintered material from the data acquired in the hysteresis and magnetization curves. For this, it was necessary to design the CAD of the engine to be simulated, create a new material in FEMM 4.2 [24], and insert the parameters collected in the magnetic tests. Figure 10 shows this procedure, in which data B (magnetic flux) and H (field strength), which correspond to the magnetization curve of the material, are inserted and saved in the program. In addition to these data, information such as electrical resistivity of the material, conductivity and other data can also be entered if necessary.

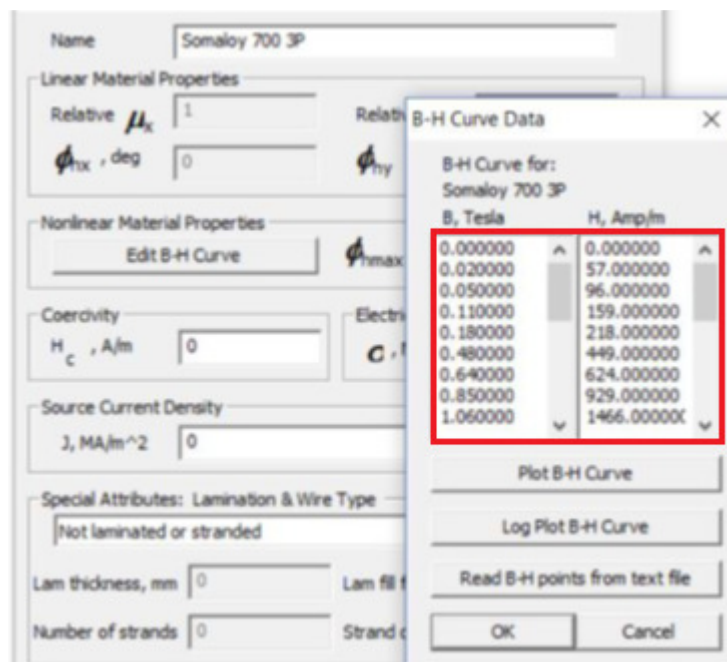


Figure 10: Insertion of the magnetic parameters of the Somaloy 700 3P material

After inserting the magnetic parameters in the software, it is possible to plot the magnetization curve (curve B/H) of the material to verify the behavior of the curve. You can do this for all materials that are collected data via curve plotter. These different graphics facilitate the analysis of the motors from the simulations, as there is no need to redo the magnetic parts (rotor and stator) of the motor again.

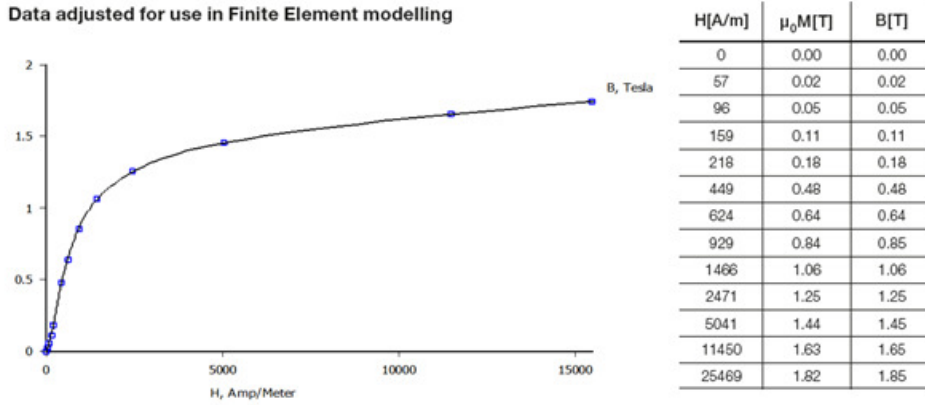


Figure 11: Plot curve B/H parameters of the Somaloy 700 3P material

4. Results

The simulation of magnetic materials helps to survey the electrical and magnetic parameters of the material in view of the laborious task of designing the specimens. In this way, the study of the magnetic behavior of an SMC from a metallographic image provides a quick analysis and allows changes in parameters in order to find an alloy composition and mechanical characteristics such as greater compaction to reduce pores.

4.1. Electromagnetic simulation of SMC materials

One of the objectives was to determine the behavior of the magnetic flux (B) for sintered composite materials. Therefore, using as an reference an image of the crystallographic structure (Figure 12.a) of one of the samples used in this study. Thus, after the image treatment and the generation of contours of each of the grains (Figure 12.b), it is possible to start the process of creating the model and later simulating this material. The grains can be seen in Figure 12.c, in green. The size of the grains varies from 70 μm to approximately 125 μm . Figure 12.d, on the other hand, shows the process of naming each area (area composed of the entire length of the contour), and the creation of the mesh (mesh) used by the finite element method of the FEMM compiler [24].

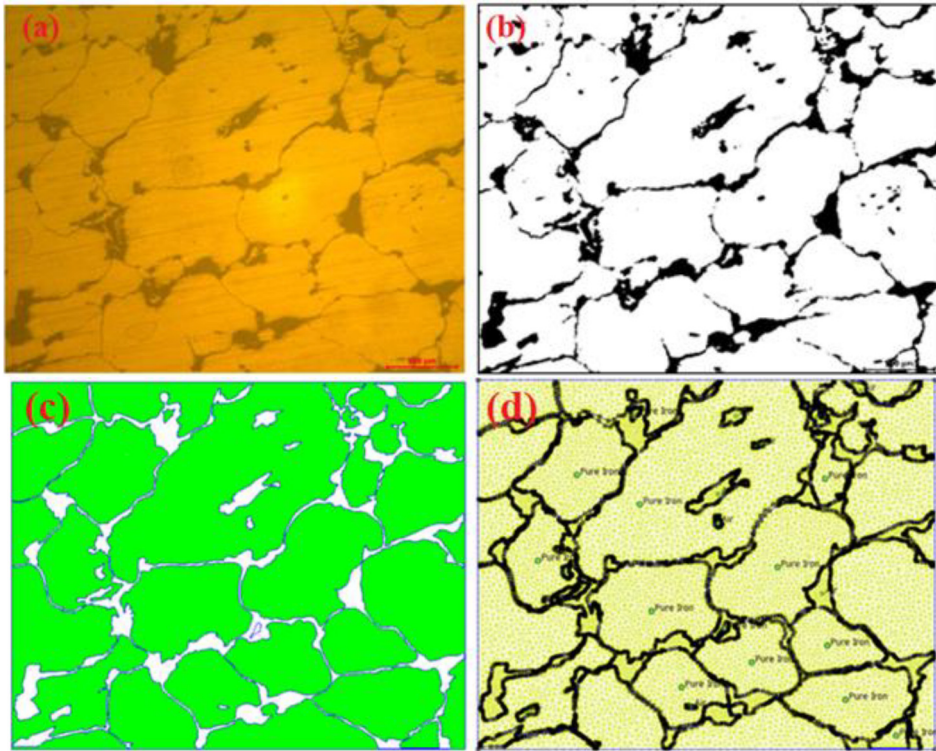


Figure 12: (a) Metallographic image of the Somaloy 700 3P; (b) binary image; (c) green grains and pores / isolation in white; and (d) the grains are named as pure iron and the pores / insulation as air; and (e) magnetic field applied from top to bottom, creating a vertical induced magnetic flux

Different parameters can be analyzed, in addition to the flow lines that are more visual. Thus, the analysis of the induced magnetic flux, in a metallographic image of the Somaloy 700 3P sample, can be seen in Figure 13.a, which shows the circulation of a magnetic flux (B) from top to bottom.

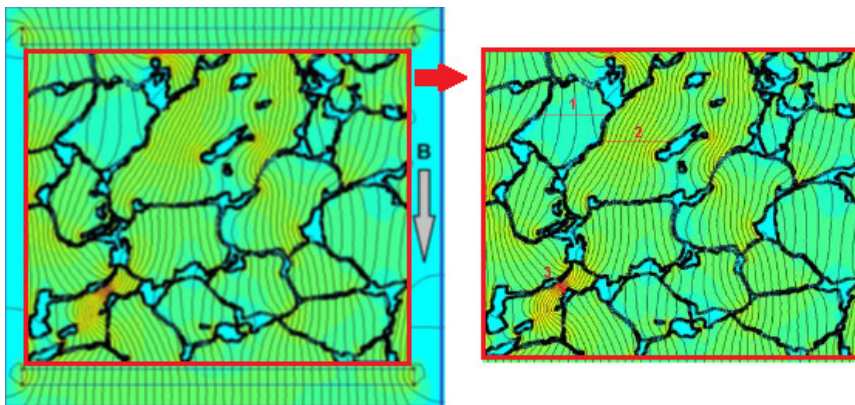


Figure 13: (a) magnetic field applied from top to bottom, creating a vertical induced magnetic flux and (b) Measuring the magnetic flux passage in different paths (grains)

Finally, in Figure 13.b, the magnetic flux intensity induced in each of the iron particles was measured. Three measurements were made, where line number 1 has a B equal to 0.19 Tesla. In line number 2, the B corresponds to 0.76 Tesla. Finally, line number 3 has the highest B value, around 1.7 Tesla, due to the drastic decrease in grain width. In some regions there is a passage of reduced magnetic flux due to the fact that the insulating layer of the grain is thicker, making the flow lines opt for other paths of less resistance. Another important factor to be observed refers to the existence of pores (holes), due to the low pressure used in the pressing of the sample.

4.2. Electromagnetic simulation of induction motors

The simulation of induction motors is intended to verify the operation of the motor without it being assembled in practice. The results obtained in the simulation, compared with the parameters already known, provide a study route in order to allow adjustments in SMC materials without the need to build a new engine.

Thus, Figure 14 shows the simulation of this three-phase induction motor, which is usually assembled with typical laminated plates (M-19 Steel). In the case of replacement of the magnetic composite materials, according to their B/H curve parameters was observed in the simulation results that the maximum magnetic density was close to 1.22 T for a peak current of 1 Ampere, being about 17 % less than the conventional model (rolled sheet).

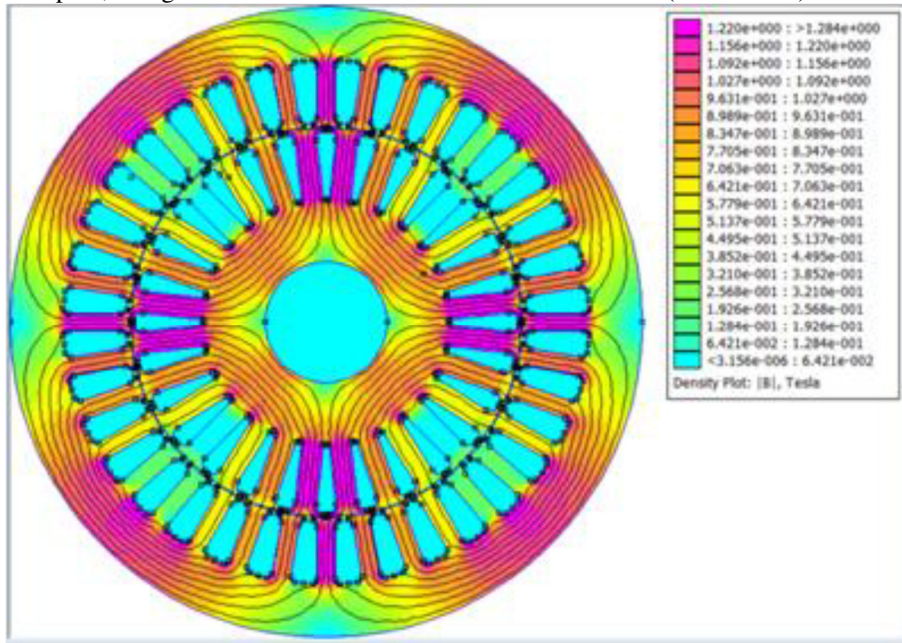


Figure 14: Simulation of a motor using the parameters of the Somaloy 700 3P

In addition to the lower magnetic flux density, SMC also had a lower magnetic permeability (ie, ability to conduct flux versus the applied magnetic field) and a considerable increase in electrical resistivity, reaching $14 \mu\Omega.m$.

From these data, a relationship between the composite and conventional materials can be made and the differences are justified by: (i) the presence of (empty) pores in the sample due to the lack of pressure at the time of compaction the sample; (ii) the insulating layer is very thick, depending on the quality of the particle covering; and (iii) o increases the electrical

resistivity of the material due to the large amount of particles that lead to a B value below the saturation value (1.22 T) as shown in lines 1 and 2 of Figure 13.b.

5. Conclusion

In this study, the electromagnetic properties of SMC materials were analyzed, both commercial and developed by the author, thus creating a simulation environment for tests on three-phase induction motors. Through the development of this work it is possible to analyze the characteristics of the soft magnetic composites, both experimentally and by simulation, of the Somaloy 700 3P alloy, from the company Höganäs AB, and of SMC samples from the author himself.

The analysis of the material sampling phase was used to survey the physical and magnetic properties, where it was possible to compare them with values obtained in the literature, either with previous works or with the material manufacturer. Subsequently, these parameters were inserted in a simulation environment and the electromagnetic behavior of the material was observed, in addition to the comparison of this alloy with conventional motors assembled from laminated plates.

The results found in the simulations show a lower performance of the Somaloy 700 3P alloy, however its use in a 3D design that increases the intensity of the magnetic field will result in a considerable increase in the magnetic flux (B), thus surpassing the rolled steel. Phosphatized SMCs, on the other hand, had less saturation magnetization, which interferes with the performance of electric motors.

Another important factor that this study presented was the analysis of the magnetic flux based on the metallographic images of the samples used in the tests. These images are essential for understanding the magnetic behavior of SMCs, as in the simulations it can be observed that variables such as particle size, thickness of the insulating layer and presence of pores can significantly affect the B/H ratio of the developed or motor part .

Finally, the work proved to be interesting with regard to computer simulations of electric motors, especially with regard to new magnetic materials, both commercial ones and the materials studied and produced by the endemic environment.

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