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Magnetic properties of iron powder sintered by medium-frequency electrical resistance sintering

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ABSTRACT

Medium-frequency electrical resistance sintering (MF-ERS) is a technique that uses the application of pressure and heat, heat coming from the Joule effect, simultaneously to metallic powders. In this study, this technique consolidates commercial iron powders, and the porosity distribution and hysteresis curves of the compacts were analysed. Compact consolidated by conventional powder metallurgy (PM) was compared with the results obtained.

1. Introduction

The advantages offered by the MF-ERS manufacturing route to magnetic products are high precision dimensions in the final form, the speed of the process, the surface finish, the absence of a protective atmosphere and the application of low pressure [1].

Direct current can be used because of medium frequency technology, and the size and weight of the sample can be reduced without changing its properties. The main objective of this study is to achieve good magnetic properties with the MF-ERS technique and to take advantage of the technique's speed and energy savings.

Therefore, the porosity distribution, microhardness and magnetic properties of the hysteresis curves of the Fe compacts achieved by MF-ERS were analysed and compared with those of a compact prepared by the conventional PM route. This study is a continuation of the work in [2], looking for typical uses taking advantage of the good fabrication characteristics of MF-ERS.

2. Experimental procedure

2.1. MF-ERS equipment

A resistance welding equipment (Fig. 1) was adapted to produce a rectified and electronically controlled current with a 1000 Hz, 100 kVA three-phase transformer. The maximum force produced is 15 kN.

The main disadvantage of this technique is that it is difficult to measure the temperature within the compact, as it is not homogeneous. The only possible way to know the temperature distribution is by means of the simulation process, which is described in [3].

2.2. Experimental procedure

The idea was first described by Lenel [4] and has been modified and developed at the University of Seville, obtaining the current procedure described in the following. MF-ERS experiments start with a cold-pressing period of 1000 ms, with no current passing through, followed by a heating period, which could be varied in each experiment. The last period is a cooling period of 300 ms with only pressure applied again. The applied pressure was always 100 MPa. Experiments were carried out with current intensities of 6, 8 and 10 kA and heating times of 400, 700 and 1000 ms, as shown in Table 1.

A mass of 3.5 g of the powder was used and poured into the 12 mm diameter die.

The electrically sintered compacts obtained were compared with compacts consolidated by the conventional powder metallurgy (PM) route. The sintering conditions of the conventional PM route were a pressure of 500 MPa, and furnace sintering for 30 min under vacuum at a temperature of 1175 °C.

HV1 Vickers microhardness, with the equipment DURAMIN-A300 (Struers, Germany), was also measured.

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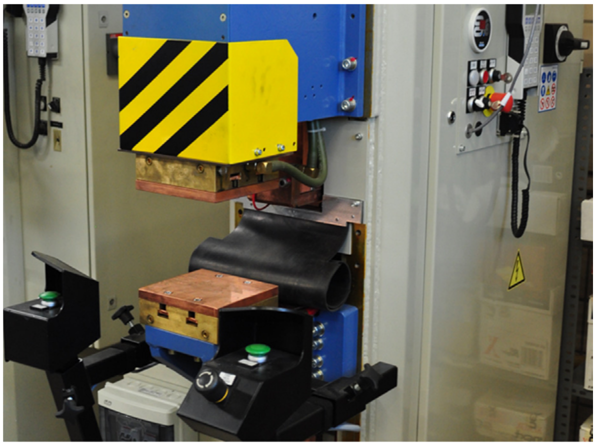

Fig. 1. MF-ERS equipment.

Table 1
Different sintering conditions.

Heating Time (ms)		400	700	1000
Intensity (kA)	6	x	x	x
	8	x	x	x
	10	x	x	x

3. Material

The powder chosen as the starting material was commercially pure iron, Fe WPL200 (QMP, Mönchengladbach, Germany). It was chosen because it is a simple material to analyse with the advantages and disadvantages it presents in the MF-ERS technique.

The mean particle size of 78 µm was measured using the laser diffraction technique (Mastersizer 2000, Malvern, UK) as shown in Fig. 2. The apparent density of 2.65 g/cm^3 was also measured.

4. Discussion

The first property measured in the MF-ERS compacts was their final porosity Θ_F. This porosity is shown as a function of the sintering conditions of current intensity and sintering time applied during the process in Table 2.

Fig. 3 shows a cross-section of the electrically sintered compacts cut to shape and Fig. 4 shows a diametrically cut cross-section of a conventionally sintered compact.

While the conventionally sintered compact has a relatively uniform porosity of about 15%, the compacts that are sintered by MF-ERS have lower porosities and, therefore, higher densities under the best conditions without having to use reducing atmospheres. However, as mentioned at the beginning, as a consequence of the different temperature distribution throughout the compact, the porosity is more heterogeneous in the MF-SRE compacts than in the PM compacts.

As can be seen in the centre of the compact, the temperature reached is higher, so the densities in this area of the compact are better than in the periphery of the compact. The fact that the temperature is higher in the centre is due to the fact that the wafers, the electrodes in contact with the plates that are cooled, and also the walls of the die cause the material in contact with them to cool much earlier.

The porosity obtained, which has suitable values for the magnetic applications of interest in this study, is achieved in the compacts with the highest sintering conditions. The 10 kA and 8 kA compacts with higher sintering times would have better results by eliminating the most superficial layer where the porosity is higher.

The microhardness of fully dense specimens was 92 ± 2 HV1, being in the conventionally processed compact of 68 ± 5 HV1. The results of the measurements of the different conditions of MF-ERS are shown in Table 3.

Lower hardness values are reached in the conventional specimen compared to the MF-ERS compacts. To compare this value, the compact with porosity like the conventional one has been

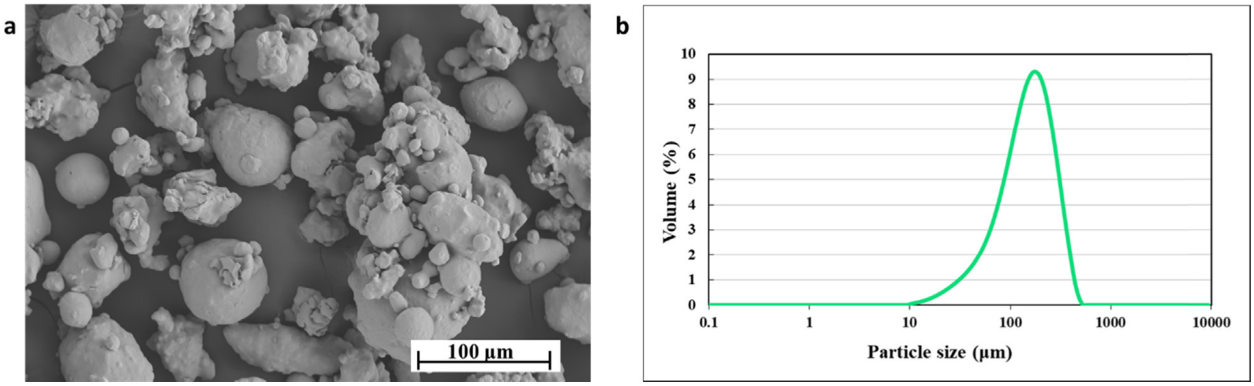

Fig. 2. (a) SEM micrograph of the iron powder and (b) granulometric curve of the powder.

Table 2
Final porosity (Θ_F) as a function of the sintering conditions.

Heating Time (ms)		400	700	1000
Intensity (kA)	6	0.30 ± 0.02	0.28 ± 0.01	0.24 ± 0.01
	8	0.19 ± 0.02	0.16 ± 0.02	0.13 ± 0.01
	10	0.12 ± 0.02	0.08 ± 0.01	0.06 ± 0.01

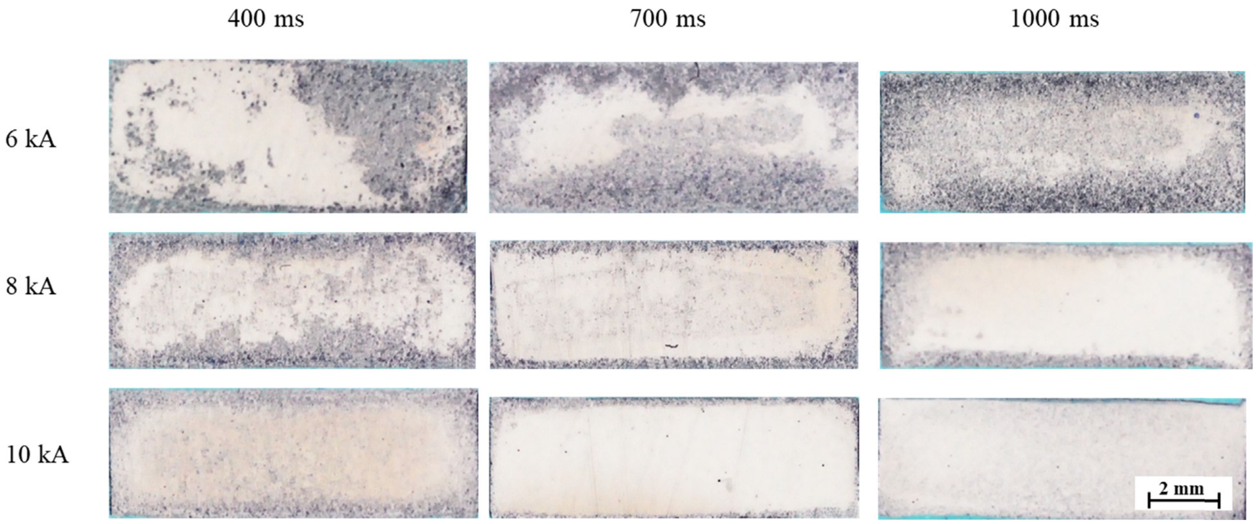

Fig. 3. Porosity distribution of MF-ERS compacts.

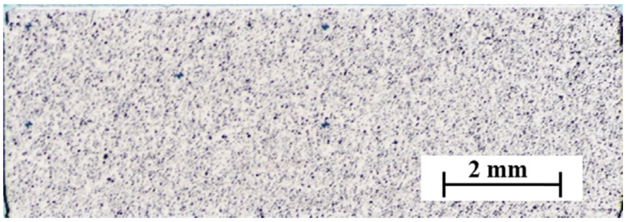

Fig. 4. Porosity distribution of PM sintered compact.

taken as a reference (8 kA and 700 ms). This could be due to the fact that the electrical sintering process achieves a better union between the particles and therefore the hardness values are higher. In addition, it is observed that the hardness increases as a function of the increase in sintering intensity and time.

To compare the magnetic properties of the MF-SRE compacts with those of the PM sintered compacts, the coercive field H_C has been measured. Table 4 shows the coercive values of the electrically consolidated compacts. The coercive value of conventional sintering is 198 ± 5 A/m.

Fig. 5 shows, as a function of porosity, the coercive field of the compacts obtained by MF-SRE and PM. The porosity present in the compacts causes the coercivity values to increase with higher porosity values [5,6]. This trend confirms that porosity has an important influence on magnetic properties [7,8,9].

This observed variation in values is not only due to porosity, but also to pore morphology and pore size, grain size, residual stress values that also affect these material properties [10–12].

The value of the coercivity reached by the sample that is more densified, and therefore has lower porosity, is 161 A/m. This value is below the value achieved by the conventionally sintered compact (198 A/m).

Therefore, the coercivity values obtained in the compacts sintered by means of MF-ERS at worst conditions are not much worse than the values obtained in the compact sintered by the conventional method (PM) and not only that, but these compacts consolidate in much less time, with lower pressure values and do not need a reducing atmosphere in the process. In fact, it is possible to reduce this value by improving densification with reducing sintering atmospheres, which is a future line of this research.

Achieving these good magnetic properties through such a fast and less energy intensive process is already a major achievement.

Table 3
Microhardness values (HV1) for different MF-ERS compacts.

Heating Time (ms)		400	700	1000
Intensity (kA)	6	37 ± 9	45 ± 8	58 ± 9
	8	61 ± 16	76 ± 9	80 ± 5
	10	81 ± 9	85 ± 5	90 ± 6

Table 4
Values of the coercive field (H_C) for each MF-ERS compact in A/m.

Heating Time (ms)		400	700	1000
Intensity (kA)	6	0.30 ± 0.02	0.28 ± 0.01	0.24 ± 0.01
	8	0.19 ± 0.02	0.16 ± 0.02	0.13 ± 0.01
	10	0.12 ± 0.02	0.08 ± 0.01	0.06 ± 0.01

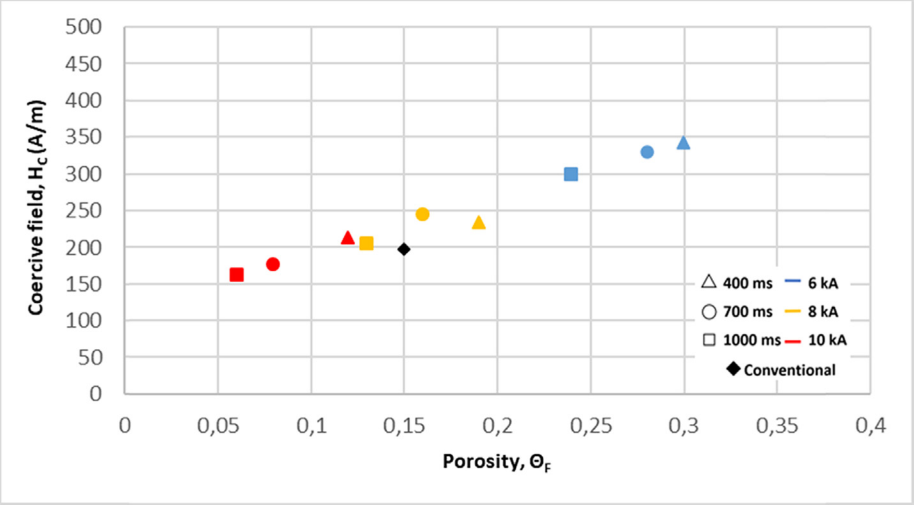

Fig. 5. Coercive field (H_C) vs. final porosity (Θ_F) or MF-ERS and conventional PM compacts.

The fact that the hardness of the compacts reaches high values means that these compacts have a greater number of applications, which means that they perform an evaluation in each particular case of the properties based on their subsequent utility.

5. Conclusions

The results obtained regarding the porosity and microhardness values of the compacts are in accordance with the processing conditions, as expected, since lower porosity and higher microhardness values are obtained for the more energetic sintering conditions.

The microhardness of the sintered materials in the oven is somewhat lower than that obtained in the electrically consolidated materials. This can be caused by the high cooling rate of the electrically consolidated compacts, a fact that produces the creation of residual internal stresses and therefore the hardness increases.

The porosity of the material has affected the coercivity of the material, as expected. It has been observed that the coercivity increased as the porosity values increased.

If we compare the coercivity values of the conventionally sintered compact (PM) with the coercivity values in the MF-ERS sintered compacts with porosity values similar to those of (PM), we observe that they are very similar values. This is a very important achievement for this electrical sintering technique.

It has been demonstrated that the MF-ERS consolidation route can be an alternative to obtain acceptable magnetic properties, resulting in a fast and low energy consuming process.

However, it could be interesting to study improving the final properties of the materials, with the possible option of considering a post-annealing treatment, as indicated in [13].

CRediT authorship contribution statement

R. Astacio: Conceptualization, Methodology, Validation, Writing – original draft, Writing – review & editing. **P. Urban:** Methodology. **F. Ternero:** Methodology, Validation. **R.M. Aranda:** . **J.M. Montes:** Conceptualization, Writing – review & editing.

Data availability

Data will be made available on request.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Raquel Astacio reports financial support and article publishing charges were provided by University of Seville.

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