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Effect of Surface Modification of Compacted Iron Powder Particles on Magnetization Processes According to the Landgraf Method of Hysteresis Loss Separation

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The aim of this work was to investigate the effect of surface modification of iron powder particles of two different size fractions in soft magnetic compacted powder on the results of direct current loss separation according to the Landgraf method. We found that the surface treatment of smaller iron powder particles leads to an increased contribution of domain wall displacements to the overall magnetization process, while the surface modification of larger iron powder particles results in an increased contribution of the rotation of the spontaneous magnetization vector to the magnetization process of the compacted soft magnetic powder.

topics: magnetic materials, magnetic hysteresis, magnetic cores, magnetic properties of magnetically ordered materials

1. Introduction

Soft magnetic materials play a key role in modern energy conversion technologies [1]. With the growing demand for environmentally sustainable solutions in energy generation, storage, and transportation, the importance of such materials is steadily increasing [2]. For practical implementation, soft magnetic materials must be formable into complex geometries, enabling the realization of three-dimensional magnetic circuit designs [3]. In direct current (DC) applications, such as the pole pieces of electromagnets, high-purity iron remains the material of choice due to its excellent magnetic permeability and low coercivity [4].

2. Principle of DC loss separation

In 1999, Landgraf's group proposed a method for separating quasi-static hysteresis losses into two components along the line of magnetic induction at maximum permeability [5]. The component below that line is denoted as the low induction loss component, where the primary magnetization mechanism is domain wall motion. The component above that line is referred to as the high induction loss component, where the dominant magnetization mechanism is the rotation of the spontaneous magnetization vector.

3. Preparation of samples

High-purity (99.98%) iron granules from Thermo Fisher Scientific Inc. (USA, product number 39708) with sizes ranging from 1 to 2 mm were milled in a planetary ball mill PM 100 (Retsch GmbH, Germany) to reduce particle size. The milled powder was subsequently sieved using 63, 125, 200, and 400 μm sieves to obtain two distinct particle size fractions, i.e., 63–125 μm and 200–400 μm. Both size fractions were annealed in argon at 400°C for 90 min after milling.

To reduce surface irregularities of the powder particles, half of each size fraction underwent a surface modification procedure, involving the use of a modified ball mill vial without milling balls, with glued-in 1000-grit sandpaper [6]. This surface modification

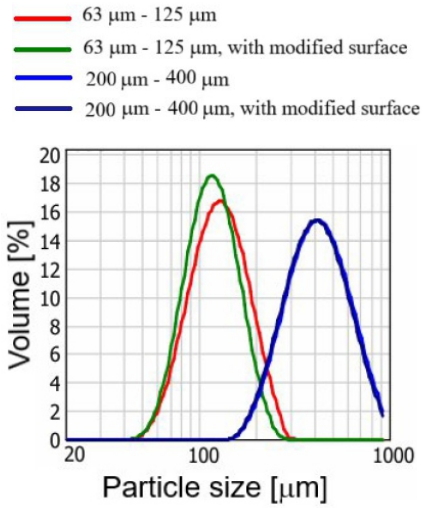

Fig. 1. Powder particle size distribution from 20 up to 2000 μm.

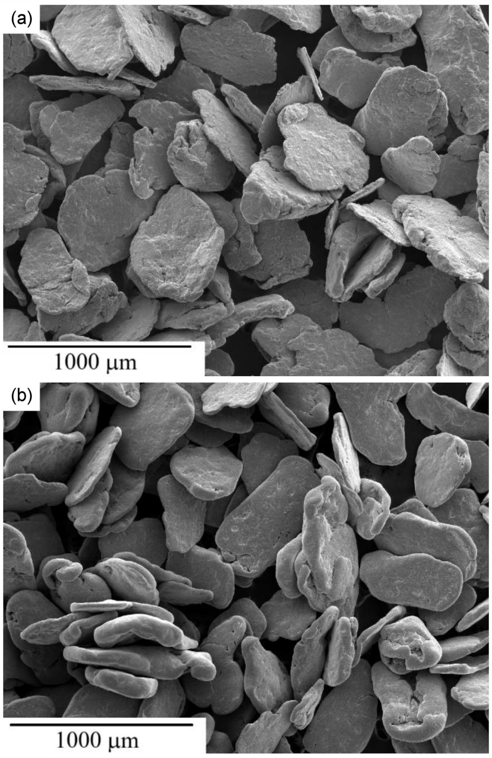

Fig. 2. Powder particles from the size fraction of 200–400 μm before (a) and after surface modification (b).

process was carried out for 70 min at 50 rpm, with a 10 s pause followed by a reversal of the rotation direction after each minute. After surface modification, the powders were sieved once again because of minor particle size reduction caused by the smoothing process. The particle size distribution of each of the four powders obtained after sieving was measured using a Mastersizer 2000M (Malvern Panalytical, NL) and is presented in Fig. 1.

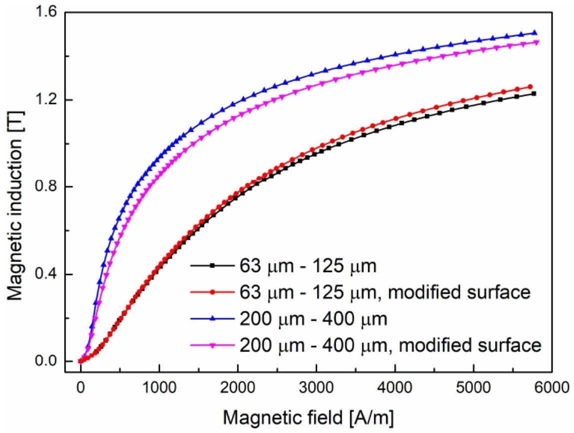

Fig. 3. Initial magnetization curves.

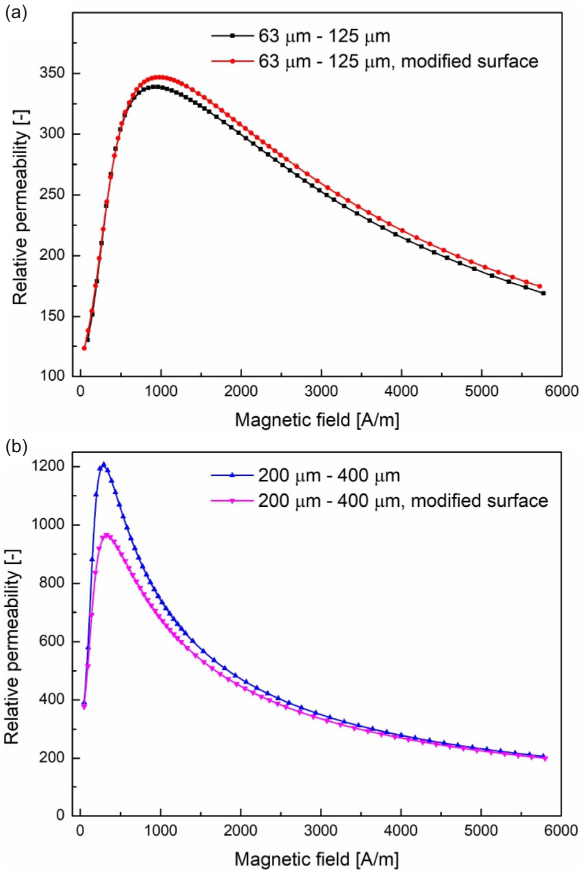

Fig. 4. Relative permeability as a function of magnetic field for samples prepared from the smaller size fraction (a) and from the larger size fraction (b) of iron powder.

The size reduction is more pronounced in the powder from the 63–125 μm size fraction due to the larger surface-to-volume ratio of smaller particles. In contrast, for the 200–400 μm fraction, the size reduction is negligible. The morphology of the powder particles was examined using a scanning electron microscope (SEM) VEGA3 (Tescan,

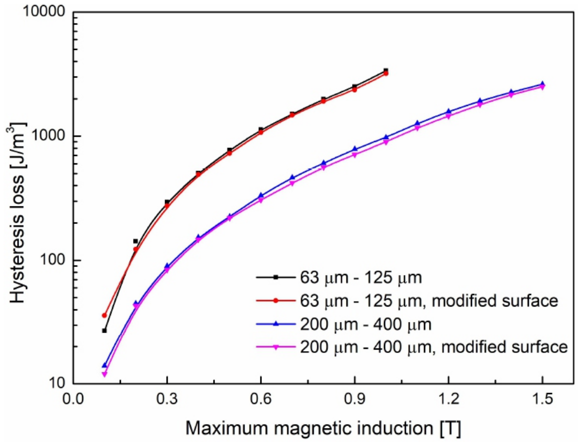

Fig. 5. Hysteresis loss as a function of maximum induction for samples prepared from both size fractions. (The y-axis is plotted on a logarithmic scale).

CZ), and a representative image for the larger size fraction is shown in Fig. 2. The powder particles that underwent the surface modification procedure exhibit a smoother shape and fewer surface irregularities.

The powders were then compacted into ring-shaped samples at 0.7 GPa and 400°C. The ring cores were subsequently annealed in hydrogen at 600°C for 1 h. Compacted cores were designated according to the sieves used for obtaining target particle sizes, with surface modification indicated accordingly: (i) 63–125 μm, (ii) 63–125 μm with modified surface of powder particles, (iii) 200–400 μm, and (iv) 200–400 μm with modified surface of powder particles.

4. Results and discussion

The initial magnetization curves, required to determine the magnetic induction at maximum permeability $B_{\mu\,\mathrm{max}}$ and the hysteresis loss with maximum induction B_{max} up to 1.5 T, were measured using an AMH-1 K-S Permeameter (Laboratorio Elettrofisico, IT).

The measured initial magnetization curves up to a magnetic field of $H = 6000$ A/m are presented in Fig. 3.

From the initial magnetization curves, the relative permeability μ_r was calculated as

$$\mu_r = \frac{B}{\mu_0 H}, \tag{1}$$

where B is the magnetic induction, and μ_0 is the permeability of free space; the resulting μ_r curves are shown in Fig. 4. The maximum permeability μ_{max} and the corresponding $B_{\mu\,\mathrm{max}}$ values are given in Table I.

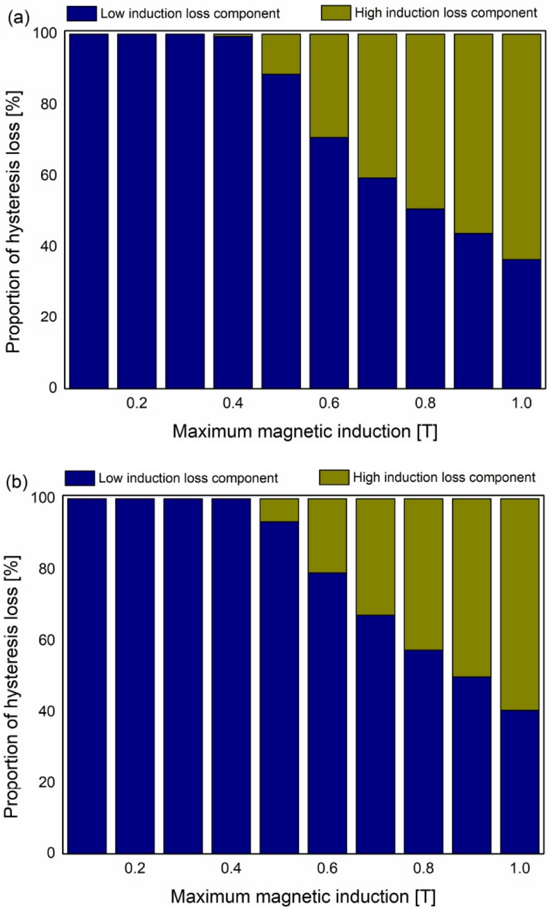

Fig. 6. Proportion of hysteresis loss for the sample prepared from iron powder with a particle size of 63–125 μm (a) and for the sample prepared from iron powder with a particle size of 63–125 μm (particles with modified surface) (b).

TABLE I

Maximum permeability μ_{max} and magnetic induction at maximum permeability $B_{\mu\,\mathrm{max}}$ of cores.

Sample designation	μ_{max}	$B_{\mu\,\mathrm{max}}$ [mT]
63–125 μm	335	385
63–125 μm, modified surface	345	430
200–400 μm	1205	445
200–400 μm, modified surface	965	400

Cores prepared from the larger particle size fraction of iron powder exhibit significantly higher values of μ_{max} compared to those made from the smaller size fraction. The effect of surface modification on μ_{max} is manifested as a slight increase for cores prepared from smaller particles and a considerable decrease for those prepared from larger particles. The value of $B_{\mu\,\mathrm{max}}$ was also influenced by surface modification, showing a change of ≈ 45 mT — an increase for the cores prepared from the smaller powder size fraction and a decrease for those prepared from the larger fraction.

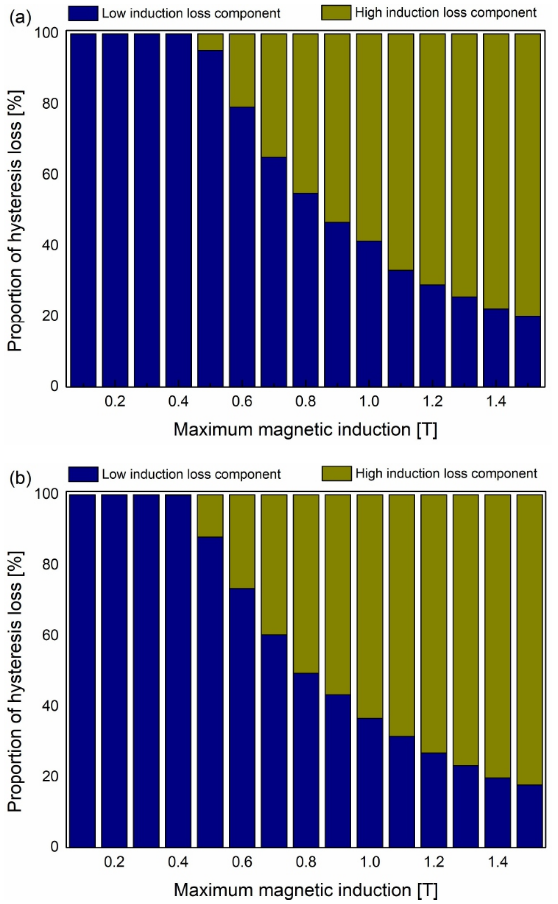

Fig. 7. Proportion of hysteresis loss for the sample prepared from iron powder with a particle size of 200–400 μm (a) and for the sample prepared from iron powder with a particle size of 200–400 μm (particles with modified surface) (b).

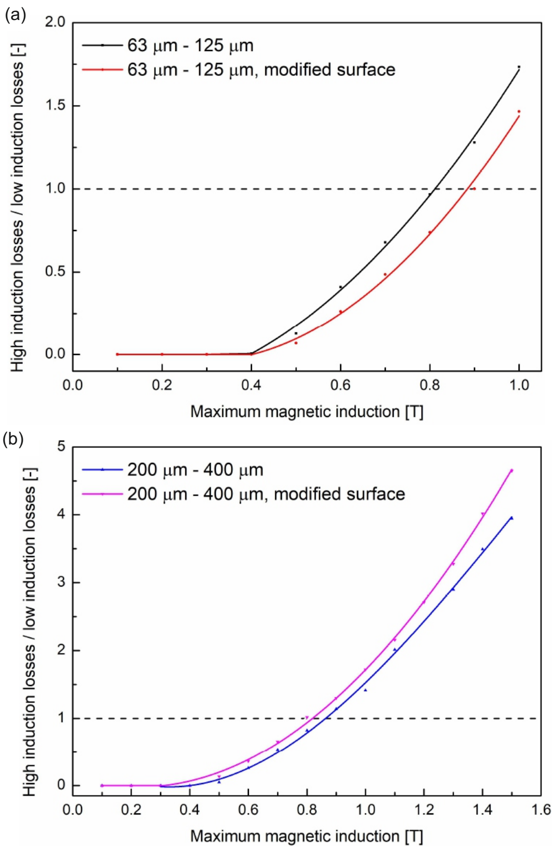

Fig. 8. Ratio of high induction losses to low induction losses as a function of maximum induction for samples prepared from a smaller size fraction (a) and from a larger size fraction (b) of iron powder.

The hysteresis loss as a function of $B_{\max}$ is shown in Fig. 5. The cores prepared from larger powder particles exhibit significantly lower hysteresis loss compared to those prepared from smaller particles. In the both size fractions, the surface modification of the powder causes a slight decrease in the hysteresis loss, except of $B_{\max} = 0.1$ T for the cores prepared from smaller particles.

The hysteresis loss was separated into a low induction loss component and a high induction loss component according to Landgraf's theory. Their respective contributions to the total hysteresis loss are shown in Fig. 6 for the cores prepared from smaller particles and in Fig. 7 for those prepared from larger particles. For all samples, up to $B_{\max} = 0.4$ T, the hysteresis loss consists solely of the low induction loss component; that is, in this $B_{\max}$ range, the dominant magnetization mechanism is domain wall motion. From $B_{\max} = 0.5$ T, the contribution of the low induction loss component begins to decrease. For cores prepared from smaller powder particles, this decrease is more pronounced in samples without surface modification, whereas for the cores prepared from larger particles, surface modification leads to a sharper decline.

To assess the relative predominance of the region where rotation of the spontaneous magnetization vector is the dominant magnetization mechanism over the region dominated by domain wall displacement, the high induction losses were divided by the low induction losses. The resulting ratios as a function of maximum induction are shown in Fig. 8.

From the graph, it is evident that surface modification of the smaller particle size fraction leads to a delayed onset (at higher magnetic induction) of the dominance of the spontaneous magnetization vector over domain wall displacement. In contrast, for the core prepared from the larger particle size fraction, the surface modification has the opposite effect, although the difference between the samples is smaller.

5. Conclusions

In this study, the effect of powder particle surface modification on the relative permeability, hysteresis loss, and magnetization processes in compacted iron cores was investigated. It was found that the surface modification of powder particles from the 63–125 μm size fraction slightly increases the relative permeability, whereas for cores prepared from powder with larger particles, surface modification results in a significant decrease in relative permeability. Surface modification leads to a reduction in hysteresis loss regardless of the studied iron powder particle size. In cores prepared from the smaller size fraction of iron powder, surface modification promotes domain wall displacement as the dominant magnetization process. While in cores prepared from the larger size fraction, surface modification increases the contribution of the rotation of the spontaneous magnetization vector.

Acknowledgments

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