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Analysis of the Preparation and Magnetic Properties of Fe/SiO$_2$ Soft Magnetic Composites

D. Olekšáková[a,*], L. Provázková[a], M. Reiffers[b],
P. Kollár[c], L. Knapčíková[a] and S. Schlögl[d]

[a]*Faculty of Manufacturing Technologies with the seat in Prešov, Technical University of Košice, Bayertova 1, Prešov, Slovakia*
[b]*Faculty of Humanities and Natural Sciences, Prešov University, 17 Novembra 1, Prešov, Slovakia*
[c]*Faculty of Science, Pavol Jozef Šafárik University in Košice, Park Angelinum 9, Košice, Slovakia*
[d]*Department Management, Communication & IT, MCI Internationale Hochschule GmbH, Universitätsstraße 15, Innsbruck, Austria*

Doi: 10.12693/APhysPolA.149.S55 *e-mail: denisa.oleksakova@tuke.sk

Soft magnetic composites are valuable materials due to their impressive magnetic properties. The aim of this research is to investigate the influence of the ball-to-powder ratio during mechanical milling and an innovative powder surface treatment on the magnetic properties of these materials. Six powder samples of pure Fe (99.98% purity) were prepared by mechanical milling with different ball-to-power ratios (BPR 3:1, BPR 6:1, BPR 9:1) and by the surface smoothing process. Magnetic properties were evaluated by measured hysteresis loops and initial magnetization curves. The results show that the best soft magnetic properties at 200 and 300 K — based on the shape of the hysteresis loops, maximum magnetization, and initial magnetization curve — were exhibited by the sample with BPR 3:1 and with powder surface treatment. The sample with a high magnetic moment is the one with BPR 9:1 and we can expect that increasing the ball-to-powder ratio causes the formation of many internal structural defects during mechanical milling, but it also creates a larger amount of small particles, which generally means smaller domain walls. We can confirm that mechanical milling and subsequently surface treatment of milled powder is a suitable precursor for preparing powdered material for further hot or cold compaction and to obtain of the required shape of soft magnetic material with excellent magnetic properties.

topics: soft magnetic composites (SMCs), powder surface smoothing, mechanical milling, hysteresis loop

1. Introduction

For large-scale industrial production, the material must demonstrate reliability in manufacturing and be easy to work with when forming cores for electronic components. Additionally, the raw material should be easily accessible and cost-effective. Crystalline Fe-based materials are highly suitable due to their excellent soft magnetic properties, affordability, and widespread use in low-energy transformer cores, electromotor anchors, and magnetic recording head cores. However, the typical forms in which these materials are prepared, such as conventional sheets or crystalline ribbons made through rapid solidification technology, are often not ideal for industrial applications. One potential solution for creating a bulk material suitable for industrial use is hot compaction of powder, which can be produced through mechanical milling of thin ribbons, powder mixtures, or sheets.

Soft magnetic composites (SMCs) have played a crucial role in the evolution of electrical machines. This article investigates the potential impact of powder particle surface treatment on the magnetic properties of the resulting composites. Nowadays, SMC materials have found applications in various industries such as automotive (as components of electric motors), powder metallurgy, computing, and the electrical industry in general (transformer cores, inductors, generators). In recent years the electronic industry, due to its rapid development, has been moving toward miniaturization, multifunctionality, high integration, and green manufacturing. Additionally, the electrical, mechanical, and thermal loads imposed in electronic applications are becoming comparatively heavier, which means that the reliability requirements for magnetic properties are continuously increasing [1, 2].

One of the prominent developments in this field is the use of Fe-based SMCs, which have generated considerable interest from both engineers and researchers. SMCs are particularly valued for their distinctive combination of properties, including isotropic ferromagnetic behaviour, high electrical resistivity, and reduced eddy current losses. Additionally, they exhibit relatively low coercivity and high saturation magnetic polarization and permeability [3–5]. These favourable characteristics are attributed to their powder core structure, where individual ferromagnetic particles are insulated from one another by an electrical insulator [6, 7]. Due to these properties, SMCs are widely used in a range of electrical equipment, including transformers, various types of electric motors, sensors, and low-frequency filters. These materials have the potential to be used in high-power applications, and to create small and precise magnetic components [8, 9].

In our study, we utilized high-energy ball milling as a practical technique, despite its tendency to introduce contaminants from the milling balls and degrade particle sphericity. Preparation of powders using planetary ball mills is highly beneficial due to their ability to achieve fine particle sizes and homogeneous mixing, simplicity, low cost and environmental friendliness. The rotating action of the ball mill ensures uniform particle size distribution and morphology, enabling better control over the properties of the final product. The result is a milled powder of the required size, which is crucial for optimizing the performance and characteristics of soft magnetic composites. Subsequently, SMCs were coated with SiO_2, an insulating material with high electrical resistance, as silicon dioxide increases corrosion resistance. Specifically, the Fe/SiO_2 SMCs were fabricated by hydrolysing tetraethyl orthosilicate (TEOS) and directly coating pure iron particles with amorphous SiO_2 layers. SiO_2 particulates can effectively weaken the corrosion process by filling in crevices, gaps, and microscopic holes on the surface of Fe particles [3]. SiO_2 is a desirable material for various reasons, such as excellent stability, strong adhesion, significant wear resistance, and high thermal resistance. SiO_2 can be readily produced via the sol–gel technique, allowing for the creation of an enhanced coating layer with regulated thickness since the precursor concentration in the solution can be easily managed [10, 11].

2. Experimental study

2.1. Mechanical milling of highly pure iron granules

High-purity iron granules (Alfa Aesar, 1–2 mm, 99.98% purity) were used in this study because of iron's excellent properties. The primary focus was to examine the effect of the surface smoothing technique, but we also analysed how different ball-to-powder ratio (BPR) values affect the final magnetic properties. Mechanical milling was chosen to obtain the desired powder fraction using the Retsch PM100 planetary ball mill (steel vial and steel balls were used). At first, the iron granules were divided into three parts and milled in a planetary ball mill with different ball-to-powder ratio values. The first portion was milled with BPR 3:1, the second portion with BPR 6:1, and the last one with 9:1. The milling time was set at 120 min, with a 10-s break after each minute of milling to stabilize the material and control the mill temperature between cycles. The speed was set at 500 rpm (revolutions per minute). Iron powder was milled for two samples at a time. The first part with BPR 3:1 was made for samples S1 and S2, the second part with BPR 6:1 was made for samples S3 and S4, and the third part with BPR 9:1 was made for samples S5 and S6. After mechanical milling, the powders were sieved using a vibrating mill with sieves. Only powder particles with smaller than 400 μm were selected to serve as SMCs powder base.

2.2. Mechanical surface smoothing process (mechanical treatment)

The sieved powder was then split into two samples with the same BPR value and processed under similar milling conditions as it was mentioned earlier. Additionally, each sample with an even number (S2, S4, S6) underwent an innovative surface smoothing process. The surface smoothing process involved mechanical treatment in the planetary ball mill without the use of milling balls. Abrasive paper (Carborundum Electrite a.s., with a grain size of 1000 p/mm^2) was affixed to the milling jar walls, and the powder was treated in this setup for 70 min under the same parameters as in the milling process (500 rpm, 10 s breaks per minute, and reverse rotation). This novel method enhances the inner structure of SMCs, potentially leading to materials with improved soft magnetic properties [12].

2.3. Insulation of the ferromagnetic powder particles

To prevent electrical contact between particles, all six samples of SMC powder underwent a special insulation process based on the Stöber method of powder particle insulation. This method is based on the principle of "wet" coating, where powder particles are coated with monodisperse silica spheres of micron size, which can theoretically create a full layer of coverage for each ferromagnetic powder particle during mixing [7, 13]. For the insulation of 10 g of ferromagnetic powder, a mixture of isopropyl alcohol (320 ml), distilled water (64 ml), tetraethyl orthosilicate (TEOS, 98%, 32 ml), and ammonia (8 ml) was used. The entire process of insulation was made by using a mixer IKA Microstar 7.5. The

TABLE I

Parameters of sample preparation.

Parameters	Name					
	S1	S2	S3	S4	S5	S6
Coating	YES	YES	YES	YES	YES	YES
Treatment	NO	YES	NO	YES	NO	YES
BPR	3:1	3:1	6:1	6:1	9:1	9:1

TABLE II

Weight percentages of iron, silicon and oxygen in SMC powders.

Chemical element	wt [%]					
	S1	S2	S3	S4	S5	S6
Fe	38.33	18.36	35.08	17.39	47.27	13.30
Si	31.20	39.59	33.79	43.18	31.51	42.60
O	30.47	42.05	35.08	39.43	21.22	44.10

velocity of the mixing process was 400 rpm and the insulation was divided into two 8-h sessions, lasting 16 h in total.

3. Results and discussion

The magnetic properties of samples S1, S2, S3, S4, S5, S6 were analysed (see Table I).

The results of the chemical analysis are shown in Table II. They indicate an increase in silicon content for the samples with mechanical surface treatment (S2, S4 and S6) compared to the iron content.

For samples made with BPR 9:1, the weight percentage of Si and O increased the most, but still the volume of weight percentage is quite the same as in the other samples with surface treatment (S2 and S4). The higher weight percentage of slicon means that there is a higher amount of SiO_2 created on the surface of these smoothed powder particles, so probably it will also lead to a more consistent insulation layer of the samples.

Hysteresis loops and initial magnetization curves were measured using a VersaLab vibrating sample magnetometer (Quantum Design) [14], and the results are shown in the figures.

Figure 1 shows the hysteresis loops of samples S1, S2, S3, S4, S5, and S6 at temperature of 200 K. It is clearly seen that all samples show soft magnetic properties. Sample S2 exhibits the highest value of the magnetic moment at the saturation, which is because we can expect sample S2 to have the fastest response to changes in the external magnetic field during next measurements compared to the other samples. Another sample with a relatively high magnetic moment value is S5, prepared with BPR 9:1, which may be due to the formation of a larger number of smaller particles and therefore also faster movement of the domain walls. The lowest values of the magnetic moment are for samples S4 and S6, and these values may have been influenced by the larger amount of non-conductive inorganic insulator on the surface of the powder particles [15].

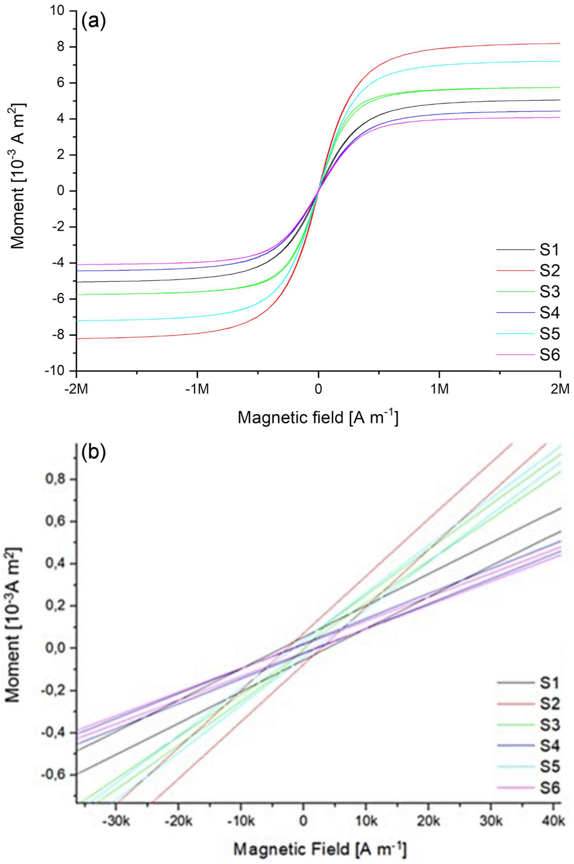

Fig. 1. Hysteresis loops of samples S1–S6 at 200 K (a) and their magnified view (b).

For samples S1 up to S6, the hysteresis loops were measured also at 300 K (Fig. 2). We can see similar results as in the case of the hysteresis loop of the samples at 200 K. We can perceive a small decrease in the maximum magnetic moment for each sample. But in the case of sample S4, even if the maximum magnetic moment is almost similar, on closer look we can see that the hysteresis loop becomes narrower at a temperature of 300 K.

Figure 3 shows the initial magnetization curves for samples S1–S6 at 200 and 300 K. The trend of these curves, as expected, follows the hysteresis loops (Fig. 2); it can be confirmed that sample S2 has the fastest response to changes in the magnetic field, and samples S4 and S6 show the lowest saturation values.

The magnetic results show that the best soft magnetic properties at 200 and 300 K, based on the shape of the hysteresis loop, maximum magnetization, and initial magnetization curve, were exhibited by sample S2 with BPR 3:1 and powder surface treatment. We can see that the insulation layer of that sample is similar to sample S1 with

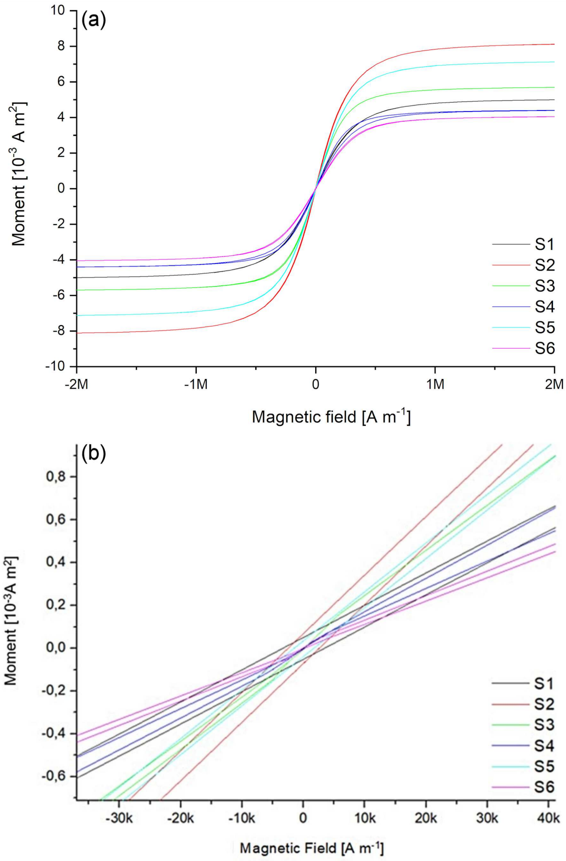

Fig. 2. Hysteresis loops of samples S1–S6 at 300 K (a) and their magnified view (b).

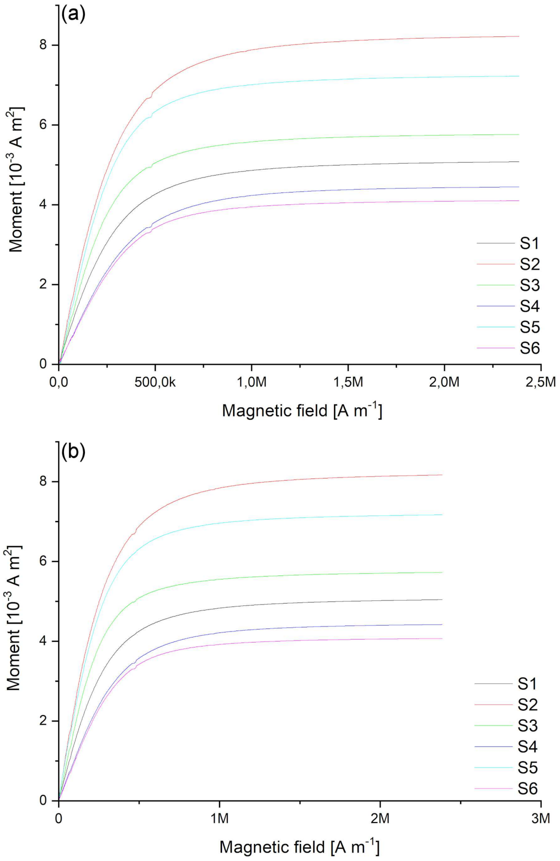

Fig. 3. Initial magnetization curves of sampels S1–S6 at (a) 200 K and (b) 300 K.

the same BPR, so we can say that the surface treatment proved its positive influence on the final soft magnetic properties of the ferromagnetic materials. Also, we can see that the sample with high magnetic moment and almost the same narrow is sample S5 which was milled using BPR 9:1, so as was expected based on a theoretic experience the increase in BPR also had a positive effect on the final magnetic properties of the material. We can also clearly see that the decrease in the maximum magnetization values correlates with the BPR in general. Sample S3 exhibit a reduction in the maximum magnetization compared to S5 (BPR 9:1), and sample S1 (BPR 3:1) exhibits an even further reduction compared to S3. Even when the insulation layer is even more consistent for samples S4 and S6 compared to all other samples, we also observe a high weight percentage of silicon for these samples. As silicon is not a ferromagnetic material and SiO_2 is an electrically isolating type of material, we can say that the amount of SiO_2 had a negative impact on the soft magnetic properties of the created composites. But it should be noticed that by controlling of the duration of the insulation process, we can decrease the amount of insulation that will be “condensed” on the powder surface; we will likely reach a smooth surface with a consistent and thin layer of insulation, which will probably have a very positive impact on the soft magnetic properties of future compacted samples.

4. Conclusions

Our investigation of the influence of BPR and powder surface treatment on soft magnetic properties yielded valuable insights:

- Based on the structure analysis, surface treatment had a positive effect on the uniformity of insulation layer.
- Sample S2, which underwent surface treatment and had BPR 3:1, exhibited the best soft magnetic properties, as evidenced by the hsteresis loop shape, maximum magnetization, and initial magnetization curve. This result suggests that surface treatment has a positive impact on the final magnetic properties of ferromagnetic materials when the amount of insulation is relatively low.
- Although increasing the BPR resulted in a high magnetic moment and a narrow hysteresis loop, as expected (see the example of

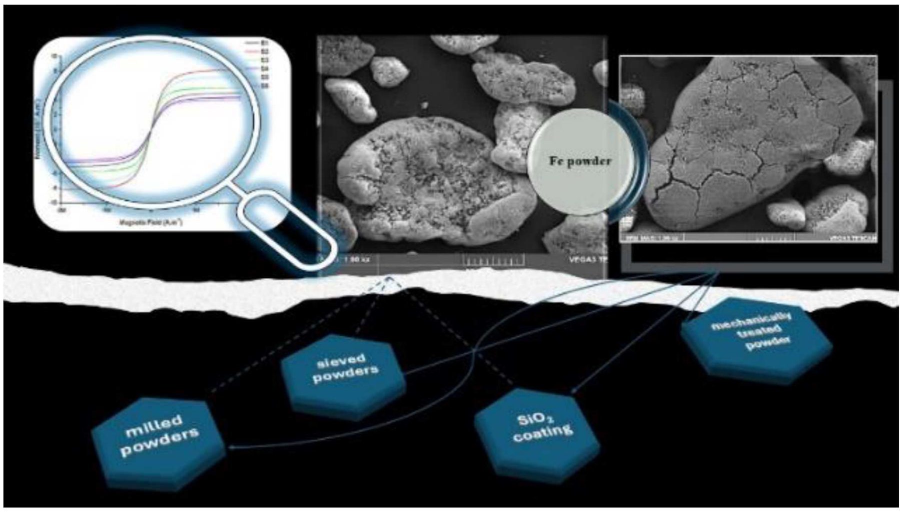

Fig. 4. Process of the preparation of samples for magnetic measurements.

sample S5 with BPR 9:1), this could ultimately lead to structural defects. However, it also resulted in smaller particles with reduced domain wall sizes, contributing to improved magnetic properties.

- The high silicon content in samples S4 and S6 likely contributed to their lower values of magnetic properties, owing to the non-ferromagnetic nature of silicon and the insulating properties of silicon dioxide (SiO_2) formed during the insulation process.

Mechanical milling and subsequently surface treatment of the milled powder is a suitable precursor for preparing powdered material for further hot or cold compaction and to obtain the required shape of a soft magnetic material with excellent magnetic properties. Powder surface treatment not only influences the uniformity of the insulation layer and its magnetic properties, but it can also potentially impact the recycling of existing composites. It can not only smooth the surface of iron powders, but also potentially remove part of the insulation layer during the recycling process of powder composites, or even remove some excess insulation of the composites. Additionally, powder surface treatment can be applied to iron powders that have already initiated oxidation process. The use of this method presents a high possibility of reducing the oxidation layer created due to improper storage of the powder materials. So, further investigation is needed to find the right balance between surface smoothness and insulation layer thickness. This includes optimizing the duration of the insulation process to achieve a thin and consistent insulation layer on a smooth surface, as well as investigating methods for removing excess insulation layers during the recycling of powder composites, hence allowing for the potential benefits of surface treatment to be realized.

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