Magnetic Properties and Morphology of Permalloy Prepared by the Compaction of the Powder

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Abstract: Permalloy is the name of nickel-iron alloys, which after heat treatment have an initial permeability much larger than that one of pure iron and are produced usually in the form of thin sheet [1]. Therefore it is logical to attempt to prepare such material in more "bulk" form, for example in the form of a cylinder or a ring, which would be more convenient for some industrial applications. The aim of this work was to investigate the influence of the powder size on ac and dc magnetic properties of the bulk samples prepared by a hot compaction of the powder.

Keywords: Soft magnetic materials, permalloy, core losses

1. Introduction

Soft magnetic materials are a well-known group of materials suitable for various ac and dc electromagnetic applications [2]. Ni-based alloys have attracted considerable attention because of their good soft magnetic properties with near-to-zero magnetostriction, low core loss and high permeability, which can be used for a variety of applications, such as magnetic recording heads and electronic sensors [3]. Polycrystalline Ni-Fe materials milled in high-energy planetary ball mills also exhibit very good properties suitable for the preparation of soft magnetic bulk materials [4, 5]. One of the ways to prepare bulk material (suitbale for some industrial applications) is the compaction of powder produced by the milling of amorphous, nanocrystalline and polycrystalline materials. The ball milling technique has been successfully used to prepare many alloys in powder form, which are suitable for compaction into a variety of shapes [6].

The aim of this work was to obtain by the analysis of the contributions of the hysteresis losses, the eddy current losses and the anomalous losses to the core losses that the fraction of all contributions strongly depends on the particle size of the powder used for preparation of the bulk material.

2. Experimental material and methods

The ribbon $Ni_{81}Fe_{19}$ (wt. %) was produced by melt spinning and then milled using a RETSCH PM4000 planetary ball mill for 5 and 10 hours (Fig. 1).

The milling was performed under an Ar atmosphere with a speed of 180 rpm at a ball-to-powder mass ratio of 6:1 in hardened steel vials. The handling of the powder was performed in a glove box. The samples were consolidated at 800 MPa for 5 min at 500 °C into discs with a diameter of 10 mm and a height of approximately 2.5 mm. An axial hole with a diameter of 5 mm was drilled into the disc to produce ring samples:

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■ *sample BR – a bulk sample prepared by the hot compaction of the small pieces (several mm2), prepared from the ribbon by breaking the ribbon by hand.*

■ *sample P5 – a bulk sample prepared by the hot compaction of the powder obtained by the mechanical milling of the ribbon for 5 h.*

■ *sample P10 – a bulk sample prepared by the hot compaction of the powder obtained by the mechanical milling of the ribbon for 10 h.*

The structure and morphology of the samples were characterised by using an SEM (Vega 5135 MM Tescan scanning electron microscope at a 20 kV accelerating voltage).

We have prepared coils with 35 primary turns and 50 secondary turns for ac and dc measurements. The magnetic induction B is sinusoidal when measuring the frequency dependent hysteresis cycle.
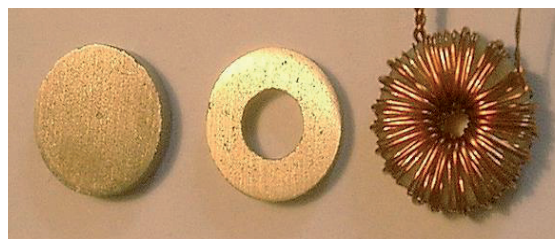

Fig. 1: *Planetary ball mill RETSCH PM 4000.*

Fig. 2: *Bulk samples $Ni_{81}Fe_{19}$ (wt.%) prepared for ac and dc measurements.*

The dc hysteresis loops at maximum flux density of 0.2 T were measured by a fluxmeter-based hysteresisgraph. The ac hysteresis loops in the frequency range from 0.25 Hz-50 Hz were measured by a fluxmeter-based hysteresisgraph with sinusoidal flux density time dependence.

The hysteresis losses Wh (dc measurements), core losses Wt (ac measurements) and coercive field were calculated directly from the measured minor hysteresis loops.

3. Results and Discussion
3.1 Structure and Morphology

The crystalline character of all bulk samples was confirmed by X-ray diffraction with Fe filtered Co-Kα radiation (Philips PW 1050) with diffracted beam graphite monochromator [7, 8].

The morphology of the surface of the Ni–Fe ribbon in as-quenched state is depicted in Fig. 3a. Small pieces of the ribbon with area of several mm2 were compacted, Fig. 3d. The ribbon pieces were brittle enough to be crushed during compaction process and the average linear size of the pieces was reduced to 5 µm. The 5 hours milled ribbon, consisting of powder elements with relatively sharp edges with size of about 30 µm (Fig. 3b), was precursor of the compacted bulk created by 3 µm elements, Fig. 3e. The powder milled for 30 hours had average size about 10 µm (Fig. 3c) and the surface of the bulk prepared from this powder is very smooth and no pores are visible, Fig. 3f [4].
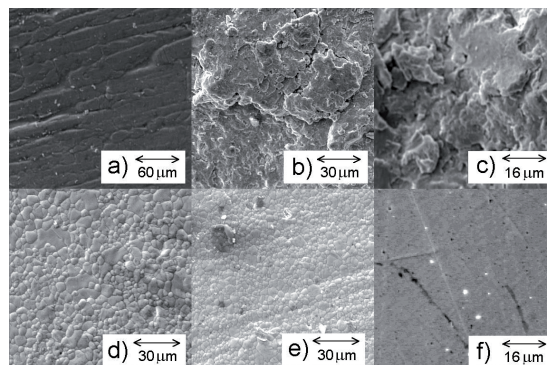

Fig. 3: *The morphology a) of the Ni–Fe ribbon's surface, b) of the 5 h and c) of the 30 h milled powder, d) of the compact prepared of broken ribbon, e) of the compact prepared from 5 h milled powder and f) of the compact prepared from 30 h milled powder [4].*

3.2 Core Losses

The core losses W (in J/m^3) in the magnetic cores can be divided into three components: hysteresis loss W_h, classical eddy current losses W_e and anomalous losses W_a [9]

$$W_t = W_h + W_e + W_a \qquad (1)$$

Hysteresis losses W_h can be experimentally determined as the area of the dc hysteresis loop.

The eddy current losses We can be expressed as

$$W_e = \frac{\pi^2 B_{max}^2 d^2}{\beta} \sigma f = C_e f \qquad (2)$$

where C_e is a constant in which are associated the material parameters and the parameters of the measurement conditions except for the frequency, d is the thickness of the sample, B_m is the maximum flux density, f is the frequency, σ is the conductivity, and β is a geometrical coefficient (dependence on the width and the height of the sample).

The anomalous losses are known to be caused mainly by domain wall branching and bowing. These losses arise from the compensation of inhomogeneous internal counterfields (caused by eddy currents) by an applied magnetic field. The anomalous losses can be expressed as

$$W_a = \frac{8.8\sqrt{S}(B_{max})^{\frac{3}{2}}}{\rho}\sqrt{G\sigma V_0}(f)^{\frac{1}{2}} = \\ = C_a(f)^{\frac{1}{2}} \qquad (3)$$

where C_a is a constant in which are associated the material parameters and the parameters of the measurement conditions except for the frequency, S is the cross section of the material perpendicular to the magnetic flux, G and V_0 are parameters that depend on the material and the magnetisation.

The dc hysteresis loops (measured at $B_m = 0.2$ T) of all the samples are shown in Fig. 4.
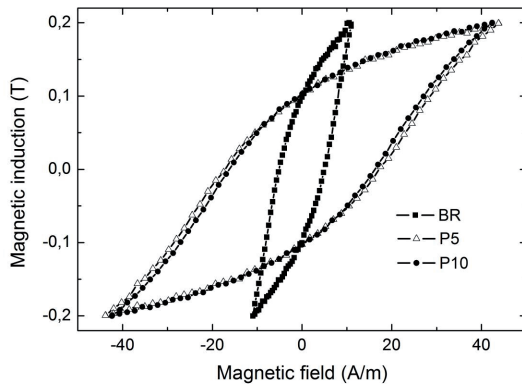
The hysteresis loop of sample BR exhibits the lowest value of the coercive field and the hysteresis losses (Tab. 1).

Table 1: *Electromagnetic parameters of the samples.*

Sample	BR	P5	P10
hysteresis losses W_h (J.m^{-3})	2.8	10.1	9.7
coercive field H_c (A.m^{-1})	5.1	17	16
C_h (J.m^{-3})	2.2	10.5	9.3
C_e (J.m^{-3}.s^{-1})	0.10	0.42	0.36
C_a (J.m^{-3}.s$^{-1/2}$)	1.3	1.1	1.9
β	19	15	14

The higher values of the coercive field and the hysteresis losses of both samples prepared by compaction of the powder (sample P5 and sample P10) can be explained by the influence of the defects introduced during the milling process, thereby creating pinning centres for domain wall displacement.

The dc hysteresis loops (measured at maximum magnetic field of 4800 A.m^{-1}) are shown in Fig. 5. The tendency to saturation for sample BR is significantly stronger than that for samples P5 and P10. We attribute this behaviour to defects introduced during the milling process for the samples prepared from the milled powder.
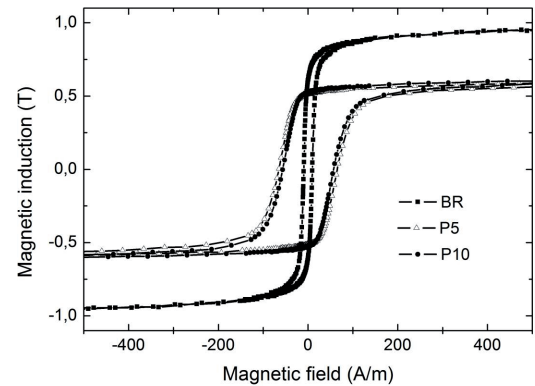

Fig. 5: *The dc hysteresis loops of samples BR, P5 and P10 measured at a maximum magnetic field of 4800 A/m.*

Fig. 4: *The dc hysteresis loops of samples BR, P5 and P10, measured at $B_m = 0.2T$.*

Fig. 6 shows the core losses as a function of frequency of all samples in the frequency range from dc to 50 Hz measured at Bm = 0.2 T. The core losses consist of all three contributions to the total

losses: hysteresis, eddy current and anomalous. This figure shows that the frequency dependence of core losses for sample P5 and sample P10 has a crossing point at f = 2.3 Hz.
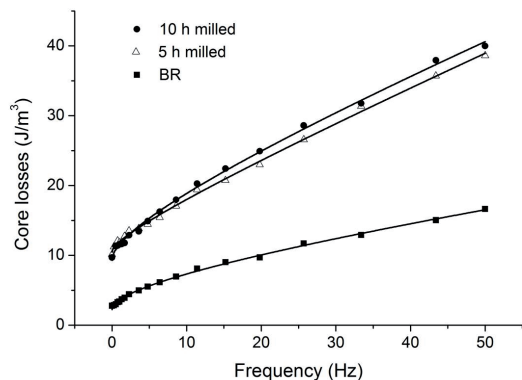

Fig. 6: *The frequency dependence of the core losses measured from dc to 50 Hz at Bm=0.2 T.*

The frequency dependences of the core losses for all samples were analyzed according to formula based on Eq. (1), Eq. (2) and Eq. (3):

$$W_t = W_h + W_e + W_a = C_h + C_e f + C_a f^{\frac{1}{2}} \qquad (4)$$

The frequency dependence of the core losses of sample BR exhibits the lowest values over the entire measured scale. This low loss is caused by the lowest contribution of hysteresis losses (likely caused by good movable domain walls), the lowest value of conductivity (responsible for the lowest eddy current losses) and the second lowest anomalous losses. This sample consists of the largest particles in which are likely present relatively large domain walls where carriers are present that are responsible for the non-negligible values of anomalous losses [10].

Sample P10 has the largest value of anomalous losses. The small particles of the powder fit very well to each other after compaction leading to the rise of the anomalous losses by restoring the magnetic "contact" between two particles that causes the increase of the effects. Anomalous losses can also be a reason for the undesirable large core losses at higher frequency [11]. We suppose that the smaller particles in sample P10 are responsible for the higher electrical resistivity (due to the higher

number of defects in smaller particles and the higher number of electric contacts in the eddy current path) leading to the lower value of the C_e coefficient in comparison with sample P5.

4. Conclusions

From the investigation of the dc hysteresis loss and the ac frequency dependence of the core losses of the compacted powder materials consisting of Ni81Fe19 (wt. %) particles of different size (5 μm - sample BR, 3 μm - sample P5 and 1 μm - sample P10) we found that the lowest value of the hysteresis losses was exhibited in sample BR, which consists of the largest particles.

The most important frequency range for the detection of anomalous losses is below 5 Hz. Omitting the frequency dependence of the core losses in this frequency range can lead to errors because of the absence of anomalous losses in the frequency range from 5 Hz to 50 Hz.

Sample P5, which consists of larger particles, exhibited larger eddy current losses than sample P10.

Sample P10, which has smaller particles, exhibited the largest value of anomalous losses due to restoration of the magnetic "contact" of two neighbours particles during compaction.

Therefore, to achieve very good soft magnetic properties of compacted-powder bulk materials, it is important to study the anomalous losses as a component of the total core losses.

5. Acknowledgments

6. References

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