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DC Magnetic Properties and Complex Permeability of Ni–Fe Based Composites

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We have investigated soft magnetic composite materials to better understand the influence of binder (resin) content on the DC magnetic properties and the complex permeability. Soft magnetic composite samples were composed from NiFe powder with phenol formaldehyde resin (ATM). Ferromagnetic magnetic powder for ring-shaped samples was obtained by milling of small pieces cut of NiFe sheets. The obtained powder was annealed to partially remove structural defects. The prepared powder was mixed with phenol-formaldehyde resin in different vol.%. The mixtures were pressed into the ring-shape samples. Complex permeability and DC magnetic properties (initial magnetization curves, anhysteretic curves and hysteresis loops) were measured. Higher inner demagnetizing fields in the resin containing samples were responsible for higher coercivity and hysteresis losses, but the permeability was stable up to much higher frequencies, compared to the sample without the resin.

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1. Introduction

Soft magnetic materials based on NiFe possess some very good soft magnetic properties as high permeability. low magnetostriction, and low coercivity. NiFe alloys in the form of thin sheets are not enable to create magnetic circuits of certain required 3D shapes, as well as are not suitable for applications at higher frequencies due to the Joule heating from eddy currents induced in metallic sheets. Soft magnetic composite materials (SMCs), consisting of ferromagnetic particles and the insulator, have some remarkable advantages over other types of materials such as sheets or ribbons, e.g. magnetic isotropy, relatively low eddy current losses at medium to higher frequencies and possibilities to create magnetic circuits in desired 3D shapes by the powder metallurgy techniques [1, 2]. The research in the field of preparation and magnetic properties investigation of SMCs strongly attracts physicists and materials scientists over the world in the last decade [1–4]. The aim of this study was to investigate the influence of different resin content in the SMC based on NiFe alloy on the complex permeability, the demagnetization factor and the hysteresis losses. The hysteresis losses (DC) are also important for AC characteristics due to low eddy currents in case of SMCs.

2. Experimental

As ferromagnetic material thin Ni81Fe19 (wt%) sheet

was used, cut into the small pieces appropriate for milling in planetary ball mill with hardened steel vials and balls. The parameters of milling were: BPR 9:1 (ball to powder ratio), 200 rpm and 6 h milling time. The obtained powder was annealed at a temperature 800 °C for 1 h in air atmosphere. The annealing was carried out to reduce mechanical stresses in particles coming from milling process. The prepared powder was mixed with ATM in different vol.% of ATM: 0, 5, 10, 20 vol.%. The obtained mixtures were pressed into the ring-shape samples at 800 MPa for 15 s. Pressed samples (5–20 vol.% of ATM) were heated up to 165 °C (limit for ATM to prevent its deterioration) for 1 h in air atmosphere, to reduce mechanical stresses induced during compaction. Different method was used to prepare the sample without ATM (labelled 0% ATM). In the first phase the temperature of powder was increased by 1 °C/min up to 400 °C, where it was held for 30 min. In the second phase the temperature was increased by $2^{\circ}C/min$ up to $1180^{\circ}C$ (sintering temperature) and held for 1 h.

Specific resistivity of 0%, 5%, and 10% ATM samples was determined by four contact method. For 20% ATM sample was chosen by direct method due to high resistance and inhomogeneity. The coercivity was measured by Foerster Koerzimat 1.097HCJ. Initial magnetization curves and DC hysteresis loops were measured by the fluxmeter-based DC-hysteresisgraph. Anhysteretic magnetization curves were measured by adapted fluxmeter-based setup [4, 5]. The nonferromagnetic components (resin+pores) of SMC was subtracted when the magnetic induction B was determined. Hysteresis loops area.

Complex permeability was measured by impedance/gain-phase analyzer HP 4194A in the

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frequency range 1 kHz–40 MHz. Real and imaginary parts of the complex permeability were calculated from the measured inductance and serial resistance of the measuring circuit and parameters of the sample and toroidal coil [6, 7].

3. Results and discussion

Table I summarizes the parameters and properties of the prepared samples, volume fractions of the constituents and pores, outer diameter D, inner diameter d, height h, mass m, specific resistivity ρ , the coercivity H_c , hysteresis losses W_{DC} at maximum induction 0.2 T and the demagnetization factor N_d .

Specific resistivity increases with the increase of resin content in composite, in case of the resin containing samples reaching values relatively high for SMCs [3], which is advantageous for AC applications.

TABLE I

Parameters and properties of SMC samples.

Sample — ATM	0%	5%	10%	20%
NiFe [vol.%]	89.61	82.39	80.30	76.42
ATM [vol.%]	0	4.33	8.92	21.19
pores [vol.%]	10.38	13.28	10.78	2.38
D [mm]	23.94	24.20	24.21	25.12
d [mm]	17.72	17.94	17.9	17.95
$h \; [mm]$	2.47	3.20	3.12	2.29
m [g]	3.38	4.14	4.01	3.35
$\rho \ [\Omega m]$	$2.22 \cdot 10^{-7}$	$7.17 \cdot 10^{-5}$	$7.48 \cdot 10^{-5}$	$1.2 \cdot 10^{-2}$
H_c [A/m]	98.2	1397	1395	1212
W_{DC} at 0.2 T $\rm [J/m^3]$	11.84	329	340	245
N_d [-]	$1.22 \cdot 10^{-5}$	$2.07 \cdot 10^{-3}$	$2.17 \cdot 10^{-3}$	$3.21 \cdot 10^{-3}$

The linear parts of the anhysteretic magnetization curves (thus for small values of B) of the ring-shaped samples are plotted in Fig. 1.



Fig. 1. Anhysteretic magnetization curves for NiFebased SMC samples with different ATM resin content.

The inner demagnetization factor N_d was calculated from the slope of anhysteretic curves for $H \rightarrow 0$ according to the expression [4, 5]:

$$N_d = \frac{1}{B/\mu_0 H_{\text{ext}} - 1},\tag{1}$$

where H_{ext} is the applied magnetic field and μ_0 is the magnetic constant. The calculated demagnetization factors N_d are in Table I.

The higher is the fraction of non-ferromagnetic component in composite sample, the more the curves are tilted, meaning the increase of N_d , as expected. (Initial magnetization curves in Fig. 2 show this tendency as well.) The inner demagnetizing fields in material containing higher amount of the non-ferromagnetic component (resin + pores) are higher. The sample 0% ATM (without the resin) is thus significantly different with much lower value of N_d compared to the resin containing samples. The heterogeneous structure of composite material consists of insulated ferromagnetic particles, often put together forming clusters, giving rise to the magnetic charges originating the inner demagnetizing fields. In SMC they come mainly from the magnetic poles on the surfaces of clusters and also from the inhomogeneous magnetization inside the clusters. The insulation of composite material as well as the pores act as gaps in ferromagnetic material, cutting-off the magnetic flux through composite sample and causing the weakening of the magnetic interaction between the particles or clusters [8].



Fig. 2. Initial magnetization curves for NiFe-based SMC samples with different ATM resin content.

In Fig. 3 DC hysteresis loops at maximum induction 0.2 T are plotted, with the corresponding hysteresis losses W_{DC} in Table I. Sample 0% ATM exhibits significantly lower W_{DC} as well as the coercivity compared to resin containing samples, due to better magnetic interaction between the ferromagnetic particles ensuring the continuity of magnetic flux.

The frequency dependences of the real and the imaginary part of complex permeability are shown in Fig. 4. For all resin containing samples the real part of complex permeability exhibits stable behaviour up to higher frequencies, near-to-constant at least up to 250 kHz. It contrasts with the sample without the resin, 0% ATM, where the real part falls steeply and from 20 kHz becomes lower than that of the sample with the 5% ATM resin content. The increase of resin content causes the decrease of the initial permeability as a result of higher inner demagnetizing fields. The relaxation frequency is attributed to the peak of the imaginary part. For 0% ATM sample it is located at low frequency before the frequency limit of the experimental setup. For the sample with 20% of ATM the peak is located at the highest frequency and with the increase of ferromagnetic content the frequency of the peak decreases.



Fig. 3. Part of DC hysteresis loops at maximum induction of 0.2 T for NiFe-based SMC samples with different ATM resin content.



Fig. 4. The real and imaginary part of complex permeability as a function of frequency for NiFe-based SMC samples with different ATM content.

4. Conclusions

The subject of this work was the soft magnetic composite materials based on NiFe alloy, and the aim was to investigate the influence of binder (resin) content on the DC magnetic properties and the complex permeability.

Inner demagnetization factor was higher for the higher resin content samples, meaning higher inner demagnetizing fields, causing the cutting-off of magnetic flux through the sample and the weakening of magnetic interaction between ferromagnetic particles. It reflected also in higher values of the coercivity and the hysteresis losses of the resin containing samples compared to the sample without resin.

On the other hand, samples containing the resin showed relatively high specific resistivity for composites, further increasing with the increase of resin content, which is advantageous for AC applications at medium to higher frequencies in order to minimize the Joule heating from eddy currents induced by time-varying magnetic flux.

Real part of the complex permeability was stable up to 250 kHz for the resin containing samples, but fell steeply for the sample without resin. With the increase of resin content relaxation frequency increased and initial permeability decreased, as a result of inner demagnetizing fields and electrical resistivity.

To sum up, the sample containing 5 vol.% of ATM resin exhibited stable permeability up to 250 kHz with the highest absolute value within the resin containing samples, and concurrently the lowest inner demagnetizing fields, while the ferromagnetic particles were found to be insulated enough, which was confirmed by the values of electrical resistivity, coercivity and hysteresis losses.

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