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METHOD OF REVERSIBLE PERMEABILITY MEASUREMENT FOR SOFT MAGNETIC COMPOSITES

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A setup for measurement of the reversible relative permeability as a function of applied magnetic field was adapted for ring-shaped soft magnetic composite samples, based on the lock-in amplifier reading of the induced voltage. Functioning of the measurement setup is described and illustrated on two types of soft magnetic composite materials: Fe-phenolphormaldehyde resin samples differing in resin content and mean particle size and FeSi-polypropylene samples with different polypropylene content. The results present the possibilities and limitations of the measurement method, together with the procedure of finding the most appropriate frequency and amplitude of AC magnetic field depending on particular material. From the measured reversible permeability the proportions of reversible magnetization processes within the total magnetization process were determined, as the ratio of reversible to differential permeability.

Keywords: magnetic measurement, reversible relative permeability, magnetization processes, composite material

1 INTRODUCTION

The reversible relative permeability measurement is a valuable tool for the investigation of reversible and irreversible magnetization processes (domain wall displacements and magnetization vector rotation) enabling to find their proportions at different values of magnetic induction as well as to determine the parameters influencing them. Studying of magnetization processes can thus provide important information on magnetic behaviour in order to improve magnetic properties of materials for particular applications. First measurements of the reversible permeability were made by R. Gans in 1911 [1]. Until now several works [2-7] dealt with the reversible permeability measurement on various bulk ferromagnetic materials *eg* iron steels. On the other hand, such measurement has not yet been shown on heterogeneous materials consisting of insulated ferromagnetic particles – soft magnetic composites (SMCs). In recent times SMCs are drawing still more attention from scientists and engineers as they possess some specific properties different from that of the other ferromagnetics [8-10]. Their advantages are *eg* magnetic and thermal isotropy, low classical losses and relatively low total energy losses at medium to higher frequencies, but non-ferromagnetic components (insulation and pores) give rise to inner demagnetizing fields within the composite material lowering its permeability [11]. In order to investigate the magnetization processes in SMCs an adaptation of the reversible permeability measurement method is needed for this kind of materials.

The aim of this work was:

- to use a known idea of the reversible relative permeability *vs* magnetic field dependence measuring in order to design the measurement setup adapted for ring-shaped samples of soft magnetic composite materials,
- to test the measurement setup on Fe-phenolphormaldehyde resin samples of different resin content and mean particle size and FeSi-polypropylene samples of different polypropylene content,
- to find the most appropriate frequency and amplitude of AC magnetic field for each sample and to specify the possibilities, limitations and accuracy of the measurement in context of soft magnetic composites features.

2 METHOD

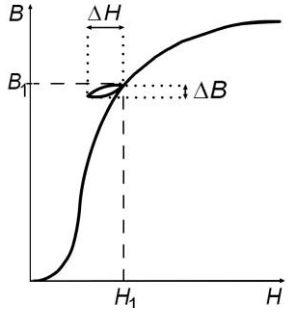

Fig. 1. Principle of reversible relative permeability measurement

2.1 Reversible relative permeability – definition

In relation to the initial magnetization curve the reversible relative permeability μ_{rev} is defined as follows: at the point of initial magnetization curve of the DC field H_1 ($[H_1, B_1]$) a small AC magnetic field with an amplitude $\Delta H / 2$ is superimposed. The material is magnetized along additional minor hysteresis loop, which is becoming a line when $\Delta H \to 0$. Then the magnetization process is realized only by reversible changes (reversible domain wall displacements and magnetization vector rotation) and the slope of the line characterizes μ_{rev}

$$\mu_{rev} = \frac{1}{\mu_0} \lim_{\Delta H \to 0} \left(\frac{\Delta B}{\Delta H} \right)_{H_1, B_1} \qquad (1)$$

Physical principle of reversible relative permeability measurement is shown in Fig. 1.

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2.2 Description of the measurement method

In general the reversible relative permeability can be experimentally obtained by variations of measurement methods based on two principles [2]: the first are ‘direct’ methods, which are based on the recording of the induced voltage when the magnetization is changing caused by changing of the applied magnetic field [1,3,4,7]. The second principle comprises methods based on measuring the inductance of a coil with the sample as a core [2,5,6], which is a function of permeability of the core material.

The amplitude of the applied AC magnetic field have to be very low in order the condition of minor hysteresis loop to become a line is fulfilled and only the reversible processes are present. For the detection of small changes in magnetization produced by very small magnetic field changes the high sensitivity instruments are needed. The accuracy of measurement is increasing with the increasing frequency of AC magnetic field (higher induced voltage), but there is an upper limit for the frequency as well – its low value is required because of the eddy current effects, enlarging the area of minor loop.

Composite materials require specific measuring conditions due to their relatively high content of non-ferromagnetic components giving rise to inner demagnetizing fields coming from the magnetic poles of ferromagnetic particles insulated from each other and thus significantly lowering the permeability [11]. In case of the reversible permeability measurement on SMCs compared to homogeneous ferromagnetics the advantage is in a little higher applied magnetic field (similar measurements were performed *eg* on steel sheets in [3]), but the much lower magnetic induction change stand for serious disadvantage.

The reversible relative permeability μ_{rev} is experimentally obtained according to its definition (Fig. 1) by a ‘direct’ method. The arrangement of the measurement setup is in Fig. 2. Functioning of the setup for measuring reversible relative permeability of ring-shaped samples as a function of magnetic field can be described as follows [10]: First the DC magnetic field is applied (primary winding on the sample is fed by four-quadrant current amplifier with maximum current of 5 A, that is controlled by the voltage source. Multimeter reads the voltage at resistor, from which the DC magnetic field value is calculated). Then the AC magnetic field of small amplitude is set (using tertiary winding, connected to the power amplifier controlled by the function waveform generator, the value of AC magnetic field is calculated from the voltage at resistor). The additional hysteresis loop is obtained. The lock-in amplifier is necessary for reading of the voltage induced in secondary winding, from which the magnetic induction is calculated. For the amplitude of AC magnetic field approaching zero, the reversible permeability is determined as the slope of the minor line (minor loop). Measurement starts from the demagnetized state up to the maximum DC field. The amplitude and frequency of the AC magnetic field is set as low as possible for acceptable accuracy of the measurement. The whole setup is controlled from PC under LabVIEW environment, via GPIB interface. The setup was designed particularly for soft magnetic composites as materials with relatively low permeability [9-11] representing limiting conditions for acceptable measurement accuracy.

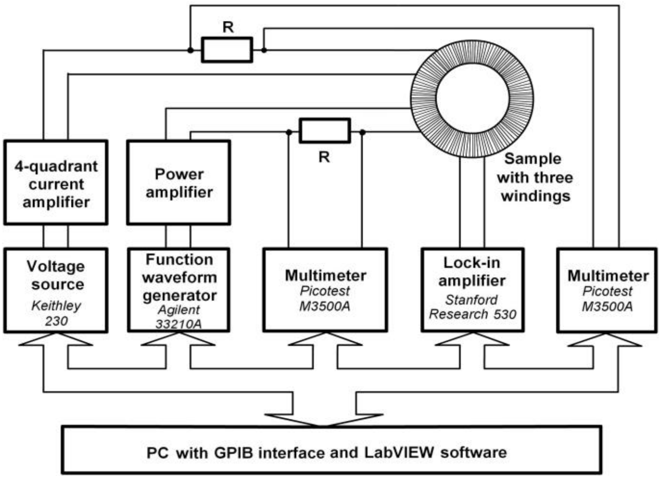

Fig. 2. Arrangement of the setup for measurement of reversible relative permeability of ring-shaped samples as a function of magnetic field

2.3 Experimental testing of the setup for reversible permeability measurement

The measurement setup was tested on two types of soft magnetic composite samples in the shape of a ring. First type was represented by the Fe-phenolphormaldehyde resin materials (iron powder ASC 100.29, Höganäs AB Sweden [12], homogenized with phenolphormaldehyde resin Bakelite ATM and compacted at 800 MPa, then cured at 165°C for 60 min in electric furnace in air) [10,11], containing 5, 10 and 15 vol. % of the resin, where the filler content of ferromagnetic component ranged from about 70 to 90 vol. % (including porosity). The samples differed in particle size distribution, as iron powder was sieved and granulometric classes with peaks at 45 μm, 75 μm, 100 μm and 160 μm were obtained (labelled F45, F75, F100, F160) [10,11]. For the second type of composites the FeSi-polypropylene samples were chosen (FeSi powder with 6.8 wt. % of Si from producer Höganäs AB Sweden [12], particle size distribution – as supplied with peak at 150 μm, mixed and injection moulded with polypropylene Moplen HP501H at 240 °C). The content of polypropylene was 30 and 35 vol. % (samples FS150-30%, FS150-35%) and the porosity was negligibly small.

For ring-shaped samples of the first type (dimensions: outer diameter 24 mm, inner diameter 18 mm, height 1.4-2.4 mm, number of turns of each of the three windings was about 300), the lowest frequency *f* and amplitude ΔH of AC magnetic field, which fulfilled the essential condition of magnetizing along small line (minor hysteresis

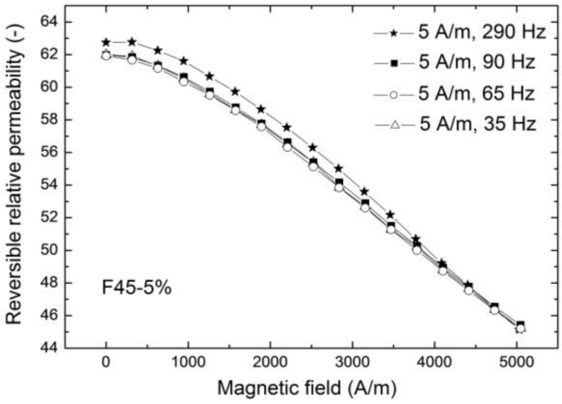

Fig. 3. Testing of reversible permeability measurement – sample F45-5%, *ΔH* = 5 A/m, frequencies 35 – 290 Hz

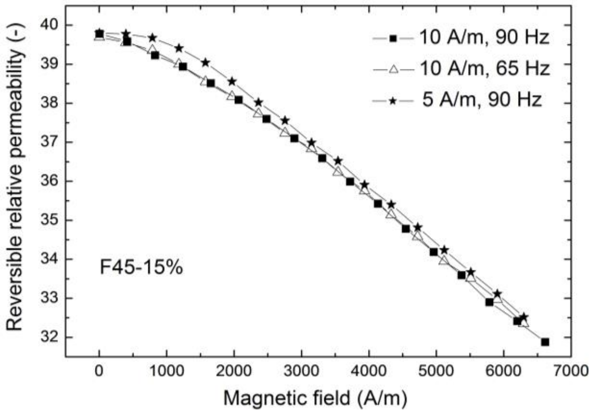

Fig. 4. Testing of reversible permeability measurement – sample F45-15%, *ΔH* = 5 A/m, 10 A/m, *f* = 65 Hz, 90 Hz

loop become line meaning only the reversible processes are present), was found from 35 Hz to 90 Hz and from 5 A/m to 10 A/m (Figs. 3, 4) [10]. (Lower values reached for higher ferromagnetic content, as the voltage induced in the secondary winding at given amplitude and frequency of the AC field depend also on the amount of ferromagnetic material and number of turns of the winding. The lowest induced voltage measurable with acceptable accuracy was in the order of 100 μV.) These measurements show accuracy within the relative standard deviations up to 2 %, but the limitation was found for the second type of samples due to their higher polypropylene content and smaller dimensions (outer diameter 14 mm, inner diameter 10 mm, height 2.0 mm, number of turns of each of the three windings was about 100) – even for the amplitude of 10 A/m and for the frequency 190 Hz the induced voltage was in the order of 10 μV (10 times lower compared to the first type) so that these measurements are not accurate (relative standard deviations more than 5 %, Fig. 5).

For the testing purpose, samples prepared by various methods were chosen and their dimensions resulted from the possibilities of those methods, as the size of the ring does not influence the measurements (numbers of winding turns were adapted to achieve the same coil constants).

In Figs. 3-5 the testing measurements are shown. We can see that μ_{rev} is decreasing with the decreasing *f* as well as with the decreasing *ΔH* (the higher *ΔH* or *f*, the higher is the value of μ_{rev}, as the area of minor loop is enlarging). The more precise measurement is the one with the lowest values of μ_{rev}, until the measurement error rises acceptably. For sample F45-5% (resin content 5 %) the different frequencies of AC magnetic field are compared (35 - 290 Hz) while the amplitude is kept constant *ΔH* = 5 A/m, Fig. 3, differences between 90, 65 and 35 Hz are not longer visible within the deviation. On the contrary, the accuracy limit for sample F45-15% (resin content 15 %) is reached at *ΔH* = 10 A/m (the measurement at *ΔH* = 5 A/m is inaccurate – that is the reason why the value is higher than for *ΔH* = 10 A/m), Fig. 4. Measurements on samples FS150-30% and FS150-35% are plotted in Fig. 5 – we can see that although the frequency and the amplitude of AC magnetic field are relatively high in the limit of the condition of magnetizing along the line, the measurement accuracy is insufficient.

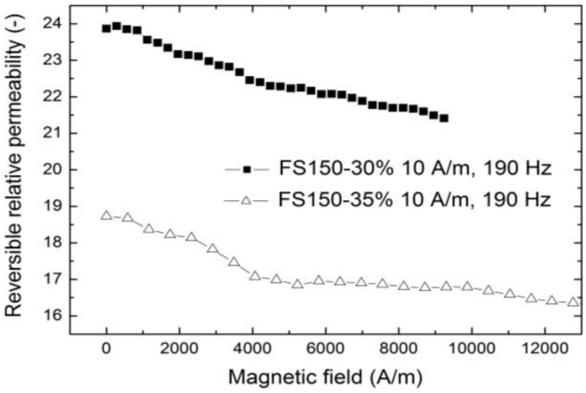

Fig. 5. Testing of reversible permeability measurement - samples FS150-30%, FS150-35%, *ΔH* = 10 A/m, *f* = 190 Hz

3 ILLUSTRATION RESULTS

The measured reversible relative permeability μ_{rev} vs. magnetic field *H* dependences of samples F100 with different resin content (5, 10, and 15 vol. %) are plotted in Fig. 6 for the illustration of the measurement setup functioning, together with the differential relative permeability μ_{diff}, which was experimentally obtained as a derivative of the initial magnetization curve at each of its points [10]. The higher the resin content, the lower the permeabilities, due to inner demagnetizing fields of particles, explained in [10,11].

It is possible to determine the proportions of reversible magnetization processes within the total magnetization process k_{rev} from the reversible and differential relative permeability measurements, as the ratio of reversible to differential relative permeability at different values of magnetic inductions along the initial magnetization curve

$$k_{rev} = \left(\mu_{rev} / \mu_{diff} \right) 100\% \tag{2}$$

In Fig. 7 the proportions of reversible magnetization processes k_{rev} at B_m of 0.1 T, 0.4 T and 0.8 T are shown for all investigated samples of the first type (Fe-phenolphormaldehyde resin composites). k_{rev} is varying with the magnetic induction, revealing the ratio of domain wall movements to magnetization vector rotation processes along the initial curve, in detail explained in work [10]. For all induction values k_{rev} is increasing with the increasing resin content and with the decreasing mean particle size [10].

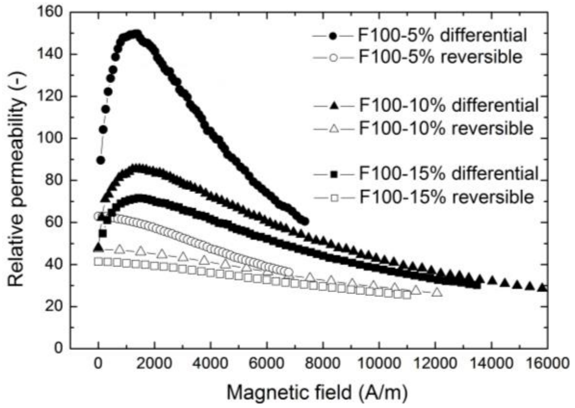

Fig. 6. Differential and reversible relative permeability vs. magnetic field dependences of samples F100 with different resin content – 5 vol.%, 10 vol. % and 15 vol. %

4 CONCLUSION

The setup for measurement of reversible relative permeability as a function of magnetic field was constructed for soft magnetic composite samples, according to the known method based on the lock-in amplifier recording of the induced voltage. Composite materials require specific measuring conditions compared to homogeneous ferromagnetics, as they contain relatively high amount of non-ferromagnetic components giving rise to inner demagnetizing fields lowering the permeability [10,11]. Reversible relative permeability was experimentally obtained according to its definition in relation to initial magnetization curve. The setup was tested on Fe-phenolphormaldehyde resin samples differing in resin content and mean particle size and FeSi-polypropylene samples with different polypropylene content. The most appropriate frequency and amplitude of AC magnetic field was found for each material (from 35 Hz to 190 Hz and from 5 A/m to 10 A/m). The possibilities and accuracy of measurement were specified in context of SMC features. From the measured reversible permeability the proportions of reversible magnetization processes within the total magnetization process along the initial curve were determined.

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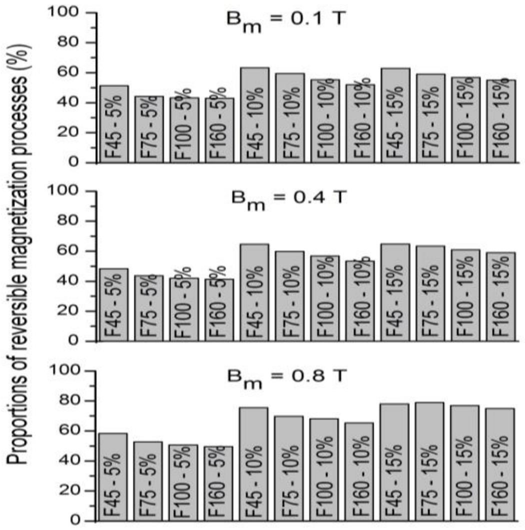

Fig. 7. Proportions of reversible magnetization processes at B_m of 0.1 T, 0.4 T and 0.8 T

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