Reversible and irreversible DC magnetization processes in the frame of magnetic, thermal and electrical properties of Fe-based composite materials

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ABSTRACT

Proportions of reversible and irreversible magnetization processes in the overall magnetization process were studied in a complex view of magnetic, thermal and electrical properties of iron–phenolphormaldehyde resin composites. They were determined experimentally at different values of magnetic induction along the initial curve. The results of total, differential, reversible and irreversible permeability measurement as well as the analysis of DC energy losses revealed the same tendencies: The numbers of movable domain walls (determining the extent of reversible processes) depend on the magnetic particle size and the resin content through the demagnetizing fields produced by the particle surfaces, lowering the interaction between particles. Thermal diffusivity was compared with Hashin–Shtrikman model indicating good insulation of particles.

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1. Introduction

Soft magnetic composites (SMCs) are a remarkable kind of soft magnetic materials composed of small ferromagnetic particles insulated from each other. They possess certain very good electromagnetic and mechanical properties e.g. the magnetic and thermal isotropy, extremely low classical losses and relatively low total energy losses at medium and higher frequencies (due to an insulating layer between iron powder particles the eddy currents are minimized), as well as a nearly netshape fabrication process ensuring a low cost mass production, which all makes them able to compete with traditionally used materials such as FeSi steels or soft magnetic ferrites at a similar production cost or even cheaper [1–5]. SMCs are well suited for use in alternating magnetic fields for electromagnetic applications such as cores with three dimensional ferromagnetic behaviour for transformers and electromotors, also as the electromagnetic circuits, sensors, electromagnetic actuation devices, low frequency filters, induction field coils, magnetic seal systems and magnetic field shielding [3–7]. One of the advantages of SMCs are their relatively low energy losses at medium and higher frequencies compared to FeSi steels – the cross-over point

is about 400 Hz, where the losses for both materials are similar (at 1.5 T of about 90 W/kg, according to [1]). SMCs are typically produced by the powder metallurgy processing methods – depending on the chosen combinations of materials and processing parameters, a wide range of properties can be obtained, so the electromagnetic and mechanical properties of SMCs can be tuned, within their physical limits, to the requirements of particular application [3–6].

The investigation of reversible and irreversible magnetization processes can provide valuable information on behaviour of magnetic materials at different magnetic flux densities or magnetizing frequencies and also finding the parameters influencing their proportions in the overall magnetization process can be of advantage enabling to produce materials with the required properties. Particularly studying the processes at DC magnetization is the starting point important also for the analysis of AC magnetization process. Up to now several works have dealt with the reversible and irreversible magnetization processes in various materials [8–11], but they have not yet been studied in SMCs by means of the reversible permeability measurement.

It is also important to study the thermal and electrical properties of magnetic materials as an energy dissipation occurs due to magnetization processes and the magnetic components of electromagnetic devices are heated through the Joule effect of eddy

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currents induced by time dependent changes of magnetic induction in material [12,13].

The aim of this work was to find:

– the proportions of reversible and irreversible magnetization processes according to the differential, reversible and irreversible relative permeability measurements,
– the relation between reversible and irreversible magnetization processes and magnetic, thermal and electrical properties as a function of magnetic induction, temperature, the mean magnetic particle size and the resin content of iron–phenolphormaldehyde resin composites.

2. Experimental details

Ring composite samples were prepared by powder metallurgy (outer diameter 24 mm, inner diameter 18 mm, height from 1.4 to 2.4 mm) for magnetic and electrical properties investigations, and thin discs (diameter 10 mm, height from 1.5 to 2.2 mm) were used for thermal properties measurements. To study the SMC properties as a function of mean magnetic particle size and resin content, the polycrystalline iron powder ASC 100.29 (Höganäs AB Sweden [14]) was sieved obtaining four granulometric classes of particle size distributions with peaks at 45 μm, 75 μm, 100 μm and 160 μm (classes labelled F45, F75, F100 and F160). For each granulometric class samples with different iron to resin ratios were prepared. Iron powder was homogenized with 5 vol.%, 10 vol.% and 15 vol.% (1 wt.%, 2 wt.% and 3 wt.%) of phenolphormaldehyde resin (Bakelite ATM) and acetone, then compacted at uniaxial pressure of 800 MPa and afterwards cured at a temperature of 165 °C for 60 min in electric furnace in air. Density and porosity were calculated from the mass and dimensions of prepared samples (density of iron: 7.851 g/cm³, density of resin: 1.39 g/cm³), reaching values about 6.7 g/cm³ (samples with 5 vol.% of resin), 6.0 g/cm³ (10 vol.% of resin) and 5.6 g/cm³ (15 vol.% of resin), porosity varied from 10% to 18%.

For the investigation of magnetic properties the initial magnetization curves were measured by the DC fluxmeter-based hysteresisgraph, from which the total and the differential relative permeability were further obtained. DC energy losses were calculated from the hysteresis loops measured by DC fluxmeter-based hysteresisgraph. Maximum induction was measured referred to the filler content of ferromagnetic material in composite sample (subtracting non-ferromagnetic components: resin and pores). The reversible relative permeability vs. magnetic field dependences were measured using the setup shown in Fig. 1. The following method was used: starting from the demagnetized state the DC magnetic field is set first, then the small AC magnetic field is applied and the induced voltage is read by the lock-in amplifier (a similar setup was used for steel sheets measurement in [11]). For the AC field amplitude approaching zero the additional small inner hysteresis loop becomes a line and the slope determines the reversible permeability.

To measure only reversible magnetization the frequency and the amplitude of AC magnetic field must be very low. The condition was fulfilled with the used values ranging from 30 Hz to 90 Hz and from 5 A/m to 8 A/m, respectively, depending on the sample resin content.

For the investigation of thermal properties the thermal diffusivity was measured by the laser flash apparatus (LFA 427, Netzsch-Gerätebau GmbH, Germany) in the temperature range from −40 to 200 °C. Cylinder-shaped samples were covered by a thin graphite layer for a good absorption of the laser beam [15]. The specific resistivity was measured by the four-contact method adapted for ring-shaped samples.

3. Magnetic properties – reversible permeability, DC energy losses and the proportions of magnetization processes

One of the key quantities characterizing the magnetic properties of magnetic materials is the magnetic permeability. In relation to the initial magnetization curve it is defined by various means. The total relative permeability μ_{tot} is calculated as

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

wherein B is the magnetic induction, H is the applied magnetic field and μ_0 is the magnetic constant. The differential relative permeability μ_{diff} comprises both reversible and irreversible magnetization processes and can be written as a sum of the reversible and irreversible relative permeability, $\mu_{diff} = \mu_{rev} + \mu_{irr}$. The derivative of initial magnetization curve (denote each its point $[H_0, B_0]$) determines μ_{diff}

$$\mu_{diff} = \frac{1}{\mu_0} \left(\frac{dB}{dH}\right)_{H_0, B_0}.\tag{2}$$

The reversible relative permeability μ_{rev} is defined as follows: at the point of initial magnetization curve with the DC field H_1 ($[H_1, B_1]$) a small AC magnetic field of an amplitude $\Delta H/2$ is applied. For $\Delta H \to 0$ the additional inner hysteresis loop becomes a line. The slope of this line, where only reversible processes are present, is of smaller angle than the slope of the initial curve at the same point. It characterizes μ_{rev} as follows:

$$\mu_{rev} = \frac{1}{\mu_0} \lim_{\Delta H \to 0} \left(\frac{\Delta B}{\Delta H}\right)_{H_1, B_1}.\tag{3}$$

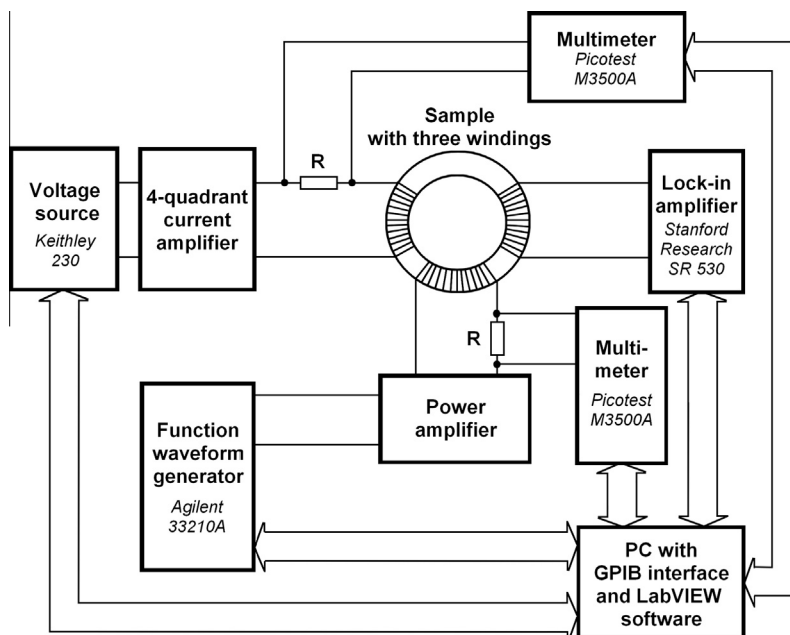**Fig. 1.** Schematic arrangement of the equipment for reversible permeability measurement.