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CHARACTERIZATION OF PERMANENT MAGNETS WITH THE PULSED FIELD MAGNETIZER

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We report on experiments aimed at precise characterization of the hysteresis loop behavior of permanent magnets by combination of different measuring methods. A discussion is made, in particular, on the application of the Vibrating Sample Magnetometer (VSM), the electromagnet-based hysteresisgraph (EMH), and the Pulsed Field Magnetizer (PFM) methods. In this framework, special emphasis is placed on advantages and shortcomings of the PFM method. Magnetic hysteresis loops have been determined on isotropic and anisotropic Ba ferrites and bonded Nd-Fe-B samples using both spherical (VSM and PFM) and cylindrical (electromagnet and PFM) samples. Overall agreement is found between the results obtained with all different measuring approaches and different sample geometries, but for a definite trend of the measured coercive field value on the impressed field rate. This can be very fast in the PFM testing (period T ~ 10 ms) and very slow with VSM testing (T ~ 1800 s), and different magnetic viscosity field contributions are consequently put in evidence. It is shown that PFM can afford both the measurement of the whole sample magnetic moment, exploiting the reciprocity principle, or the conventional fluxmetric measurement in practically sized cylindrical samples. In the latter case, non-homogeneous demagnetizing effects, though accounted for by calculation, can represent a major source of uncertainty when using low aspect ratio test specimens.

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1. Introduction

The full characterization of permanent magnets requires that a saturating field is applied before bringing the material in the second quadrant along the demagnetizing curve and determining there the remanence, the coercive field, and the energy product. With the modern rare-earth based magnets, the field strength available from electromagnets may be insufficient to achieve saturation and, in some cases, even to bring the material back to the demagnetized state, making the test technique recommended by the International Standards not applicable or, at least, little accurate [1]. On the other hand, continuous techniques based on the use of superconducting magnets are too expensive and unpractical from an industrial viewpoint. For this reason, substantial efforts have been directed in recent years at the use of pulsed field sources [2] – [4]. In these devices, peak field strengths of several MA/m can be obtained either as transient or oscillating pulses, allowing one to traverse the hysteresis loop close to saturation. Several problems however arise when precise measurements are required. On the one hand, we have to deal with dynamic measurements, an inconvenient approach when the tested materials have metallic conductivity. On the other hand, we have to face the difficulties posed by measurements in open samples. Conventional cylindrical samples can, for example, be affected both by eddy current phenomena and non-homogeneous demagnetizing fields.

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In this paper we address the problem of Pulsed Field Magnetometer (PFM) measurement of hysteresis in permanent magnets through experiments in different materials and with different sample geometries. We discuss, in particular, measurements performed up to about 6 MA/m applied field value in isotropic and anisotropic hard ferrites and in Nd-Fe-B compounds, tested either as small spherical samples (2.5 - 4 mm diameter) or conventionally sized cylinders (diameter 10 – 20 mm, height 5 – 25 mm). In the first case, we measure the magnetic moment of the point-like sample, endowed with uniform demagnetizing field, directly from the flux linked with the sensing coil, applying the reciprocity principle. With cylindrical samples, neither magnetization nor demagnetizing field are uniform. The measurement of the magnetic induction is therefore restricted to the median cross-section of the sample and the demagnetizing field is computed by Finite Element Method analysis. Comparisons are made with measurements performed, where applicable, on the same samples with the conventional Electromagnet Hysteresisgraph (EMH) and Vibrating Sample Magnetometer (VSM) methods. The rate of change of magnetization is particularly fast in PFM experiments, orders of magnitude larger than with the VSM and EMH methods, and differences in the determined coercivity values can correspondingly be observed. They are attributed to magnetic viscosity effects, as confirmed by aftereffect measurements. It is stressed that PFM measurements on cylindrical samples, preferable to the small spherical samples from an industrial viewpoint, require significant corrections for demagnetizing effects and the ensuing uncertainty may be substantial. This may compound with the effect of additional dynamic energy losses when conducting materials (e.g, Sm-Co and Nd-Fe-B sintered magnets) are to be tested.

2. Experimental procedure

The employed PFM setup was built around a capacitor bank of 3600 μF, which could be charged up to a maximum voltage of 3 kV and discharged into a coil of inductance L = 0.85 mH, provided with a 50 mm diameter 170 mm long bore. The setup could be adjusted to either oscillatory or non-oscillatory transient discharge modes, by which a maximum field of 6.2 MA/m was produced, with oscillation period ranging between 11 ms and 4.5 ms. The axial and radial dependences of the magnetic field in the bore were determined, under AC supply, by means of a small search coil, which was displaced along orthogonal directions. The test specimen and the detecting coils are arranged in the solenoid bore as schematically shown in Fig. 1. A compensated pickup coil arrangement is used for the measurement of the magnetic moment of small spherical samples. It is made of a centered 6.3 mm diameter 15 mm long coil, holding the sample at its center, connected in series opposition with one or two compensating coils, axially placed at a distance where they do not intercept the stray field emerging from the sample. This is gently glued on top of a nylon screw, keeping it firmly in place during the field transient. The achieved compensation factor is higher than 10^3, and the residual background signal is eliminated by repeating the measurement without the sample. The sample polarization $J(t)$ is then related to the measured flux difference $\Delta\Phi(t)$ by the equation $J(t) = \mu_o \frac{\Delta\Phi}{kV}$, where k is the constant of the sensing coil and V is the sample volume. The constant k is experimentally determined by emulating a magnetic moment by means of an AC supplied 3.5 mm diameter coil with calibrated turn-area product $N_2 a_2$, placed at the center of the sensing coil, and measuring the mutual inductance $M_{12} = kN_2 a_2$. Alternatively, the polarization at 400 kA/m of a soft ferrite sphere, previously determined in conjunction with a pure Ni reference sphere in a VSM setup, is measured in the PFM. The resulting uncertainty on the PFM measured absolute polarization value, allowing also for a ± 0.3 mm fluctuation in sample position upon successive trials, uncertainty on the material density, and noise, is estimated to be lower than ± 2 %. The applied field H_a is separately detected by means of few-turn axial winding, placed in a position immune from the sample-generated field and in a known relationship with the field at the center of the solenoid. The turn-area of this coil is calibrated using a reference Helmholtz source.

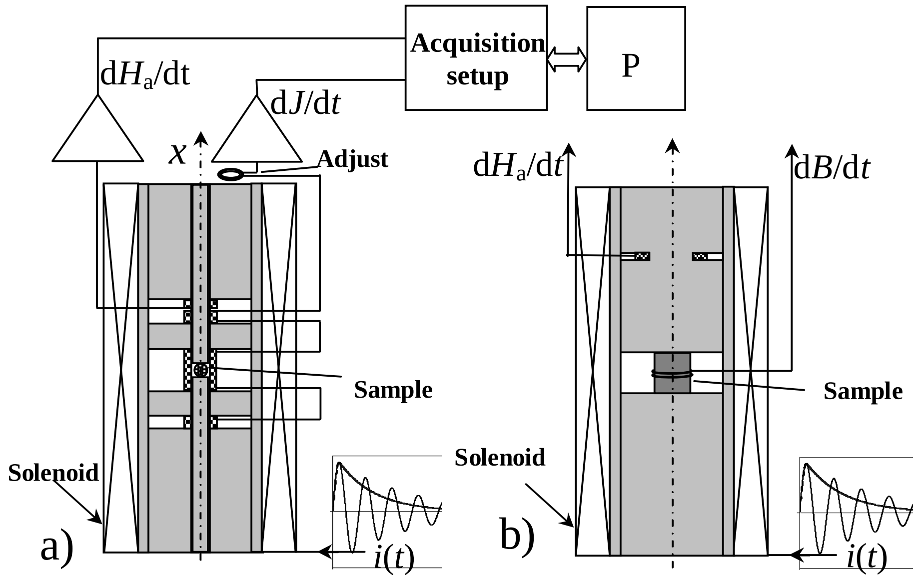

Fig. 1. Pulsed Field Magnetizer setup employed in the present experiments. A 170 mm long 50 mm diameter solenoid, supplied in the oscillating transient mode, generates a maximum peak field value $H_{ap} \cong 6\times10^6$ A/m. In the arrangement shown in a), the magnetic moment of a small spherical sample (diameter 2.5 mm – 4 mm), placed at the center of the solenoid, is detected by means of a compensated coil. The field is detected by means of a separate axial coil, held at distance where it does not intercept the stray field generated by the sample. In b) the induction at mid-section in a cylindrical/parallelepipedic sample is detected using a localized winding. The average demagnetizing field over the measuring cross-section is numerically calculated. Low-noise pre-amplifiers and a 14-bit VXI acquisition setup are employed for signal treatment.

The testing assembly for cylindrical samples of technical size is schematically shown in Fig. 1b. The induction B is measured here in correspondence of the sample mid-plane by means of a localized winding and H_a is again detected using a separate axial coil. The problem with cylindrical samples is the presence of an inhomogeneous demagnetizing field, which may give rise to a correspondingly inhomogeneous magnetization, along both the axial and the radial directions. A further difficulty may arise in metallic samples, because of the presence of eddy currents at the resulting magnetization rates, with their additional losses and possible lack of flux penetration. A rough calculation in sintered Nd-Fe-B cylindrical specimens (resistivity $\rho = 142\times10^{-8}$ Ω m) provides, for an oscillation frequency of 100 Hz and relative permeability $\mu_r = 10$, a penetration depth $\delta \approx 20$ mm. We can also crudely estimate the extra eddy current losses per unit volume at a given frequency f and peak induction B_p in a cylindrical sample of diameter D by the classical expression $W_e = \frac{\pi^2 D^2 B_p^2}{16\rho} f$. This is a lower limit estimate, formulated for a completely homogeneous magnetization process and sinusoidal time dependence of the induction. In a 25 mm diameter Nd-Fe-B cylindrical sample it provides, for f = 100 Hz, $W_e \approx 5.5\times10^4$ J/m^3, roughly amounting to an extra contribution to the measured coercivity $\Delta H_{cJ} \approx \frac{W_e}{4J_p} \approx 7000$ A/m.

The detected signals, proportional to the derivative of the applied field dH_a/dt and the polarization dJ/dt (small samples, Fig. 1a) or the induction dB/dt (localized winding on cylindrical samples, Fig. 1b) are supplied to a 14 bit 800 ksample/s VXI acquisition setup operating in a VEE software environment, by which the magnetization curves and the associated parameters are determined. In order to retrieve the polarization J and effective field H from the measured quantities B and H_a in the cylindrical samples, the average demagnetizing field $< H_{dem} >$ over the measuring cross-section must be calculated. This can be done either exploiting available tabulations reporting the dependence of the fluxmetric demagnetizing factor $N_d^{(f)} = < H_{dem} > / < M >$, where $< M >$ is the average magnetization over the mid-section, [5] or, as done in the present case, by direct Finite Element Method analysis of the magnetic field distribution. This analysis puts in evidence a relatively strong inhomogeneity of the demagnetizing field in the radial direction, which may affect the measuring accuracy when the aspect ratio of the sample is low and the material susceptibility χ is not negligible. If we take the value of $N_d^{(f)}$ calculated under the hypothesis $\chi = 0$ as representative, we obtain J and H from the measured quantities B and H_a as

$$J = \frac{B - \mu_o H_a}{1 - N_d^{(f)}} \qquad H = H_a - \frac{N_d^{(f)}}{\mu_o (1 - N_d^{(f)})} (B - \mu_o H_a) \tag{1}$$

$N_d^{(f)}$ may attain relatively high values in ordinary cylindrical samples and the accuracy of the correction is, according to Eq. (1), detrimentally affected. For example, a 1 % uncertainty in the value of $N_d^{(f)}$ in a 5 mm high 20 mm diameter cylindrical specimen leads to a 2.5 % uncertainty in the calculated J value.

3. Results and discussion

We have performed measurements with the previously described PFM method in isotropic and anisotropic Ba ferrites and in bonded Nd-Fe-B samples and we have compared them with measurements taken on the same samples with the VSM and the EMH methods. Fig. 2 provides an example of (J, H) hysteresis loops determined in isotropic Ba-ferrite cylindrical samples using the assembly shown in Fig. 1b (T = 11 ms) and applying Eq. (1) on the measured quantities B and H_a. The samples have different heights h and the same diameter D = 20 mm. The calculated $N_d^{(f)}$ values vary in the range $0.182 \le N_d^{(f)} \le 0.610$ on passing from h = 25 mm to h = 5 mm. Fig. 3 shows a comparison of loops obtained on the same material by PFM on cylinders and measurements performed by means of: 1) PFM on a 3.8 mm diameter sphere (Fig. 1a); 2) VSM on the same sphere (T = 1800 s); 3) EMH on a cylinder (D = 20 mm, h = 15 mm, T = 150 s). It is apparent here the systematic difference of the hysteresis loop width observed when applying the fast (PFM) and the slow (VSM and EMH) field rate methods, the latter providing, in particular, a slightly decreased (~ 20 kA/m) coercive field value H_{cJ}. Given the near insulating properties of the Ba ferrites, such a difference is ascribed to the thermal fluctuation aftereffect (magnetic viscosity) [6]. Magnetic viscosity is ubiquitous in hard magnets. It is due to the occurrence of thermally assisted microscopic magnetization processes, which can be phenomenologically looked at by considering, in addition to the external field H, a time-dependent random internal field, the viscosity field $H^*(t)$, aiding H in driving the magnetization reversal. The viscosity field (or better, the envelope of its peak values) is assumed to evolve with the time t spent from the start of the experiment as $H^*(t) = H_f \ln(1 + t/\tau_o)$, where τ_o, the inverse of an attempt frequency for the overcoming of energy barriers, is estimated to be around 10^{-11} s. It is apparent that the role of $H^*(t)$ may be remarkably stronger, and the observed coercivity correspondingly lower, along the VSM and EMH experiments than with the much faster PFM. The constant H_f is related to the so-called viscosity coefficient S, the quantity involved in the description of the logarithmic time decay of the magnetization displayed by a magnet when it is kept under a constant applied field. One finds that the rate of change of the magnetization can be described in this case as $dM/dt \approx -S/t$. The theory shows that $S = \chi_{irr} H_f$, where χ_{irr} is the irreversible susceptibility [7]. We have performed time decay experiments on the present Ba ferrite samples providing $H_f \sim 10^3$ A/m. Coercive field differences estimated from the predicted value of the viscosity field $H^*(t)$ for $t = T/4$ correspond within a factor 2 to the measured ones.

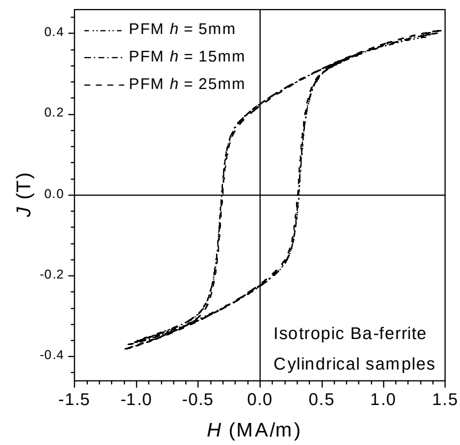

Fig. 2. Hysteresis loops in isotropic Ba-ferrite cylindrical samples of diameter D = 20 mm and different heights taken with the PFM arrangement shown in Fig. 1b (T = 11 ms). Polarisation J and effective field H are obtained from the directly measured induction B and applied field H_a after calculation of the demagnetizing coefficient and use of Eq. (1).

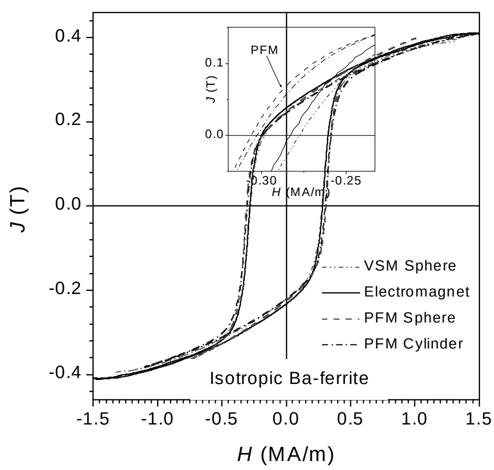

Fig. 3 – Hysteresis loops in isotropic Ba-ferrite magnets measured by means of: 1) PFM on cylinders (D = 20 mm, h = 25 mm); 2) PFM on spheres (D = 3.8 mm); 3) VSM on spheres (D = 3.8 mm); 4) EMH on cylinders (D = 20 mm, h = 15 mm). The magnetization periods are: T = 11 ms (PFM); T = 150 s (EMH); T = 1800 s (VSM). The inset provides details on the passage through the coercive field H_{cJ}.

Hysteresis loops have been measured with the three different methods here discussed also in anisotropic Ba-ferrite spherical and cylindrical samples. They reproduce to a good extent the results reported in Figs. 2 and 3, in particular the coercivity differences arising from different magnetic viscosity field contributions. Full testing of Nd-Fe-B compounds is possible instead only using the PFM, because of the obvious field amplitude limitations of the electromagnet sources employed in VSM and EMH. With the latter, however, the demagnetization curve in the second quadrant is equally obtained after exposing the cylindrical sample to non-oscillating transient in the PFM solenoid at the maximum available peak field of 6.2 MA/m. After the discharge, the sample is brought in the gap of the demagnetized electromagnet and it is inserted in the search coil, which is kept axially centered by means of suitable holder. Immediately before insertion of the sample in the coil, acquisition of the flux derivative and the field (via Hall probe) starts. It continues over the successive steps of the process, which consist in the closure of the pole pieces over the sample, the application of maximum field in the same direction of the magnetization in the sample (the "forward direction"), and recording of the so obtained peak magnetization value. The magnetizing current is then decreased to zero and reversed, to cover the whole second quadrant. Fig. 4 compares the demagnetization curve obtained in this way in a bonded Nd-Fe-B cylinder (D = 11.2 mm, h = 12.5 mm) and the hysteresis loop resulting from a PFM measurement. It is noticed in this case a faint difference only of coercivities, indicating small magnetic viscosity.

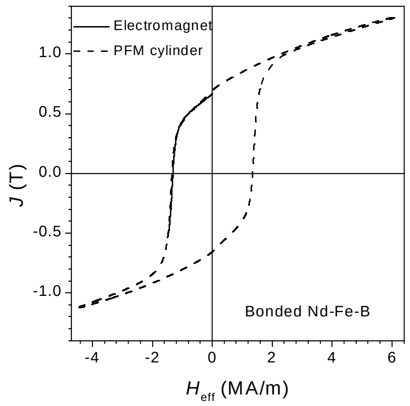

Fig. 4. Hysteresis loop measured in a Nd-Fe-B bonded cylindrical sample by PFM at maximum field 6.2 MA/m. Comparison is made in the second quadrant with the demagnetization curve obtained with the EMH method, after having subjected the sample to a saturating field in the pulse magnetizer.

4. Conclusions

The Pulsed Field Magnetizer method can afford a simple and quick characterization of permanent magnets, including those materials, like the rare-earth based compounds where the conventional methods using electromagnets as field sources fail to provide complete information. In this paper we have discussed the basic arrangements and measuring procedures by which hysteresis loops are obtained using the PFM method and examples are reported concerning a device used in the oscillatory transient mode up to maximum field of 6.2 MA/m. Distinction has been made between the classical fluxmetric approach on cylindrical samples and the approach based on the measure of the magnetic moment in small spheres. The main sources of uncertainty are identified with the demagnetizing field correction in the first case and the compensation for the large background signal in the second. Magnetic viscosity, which affects the measured coercivity value, may also be put in evidence when comparing the hysteresis loops obtained by PFM and conventional slow field rate methods, like the Vibrating Sample Magnetometer and the Electromagnet Hysteresisgraph methods. In the rare-earth based magnets additional effects related to eddy currents may occur, but they are expectedly negligible when using typical small (few mm size) spherical samples.

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