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Implementation of an automated FORC analysis and anisotropy measurements in a homemade VSM

P.N. Granell, M.R. Pagnola, L.G. Pampillo, and F.D. Saccone
Laboratorio de Sólidos Amorfos, Departamento de Física, Facultad de Ingeniería, Universidad de Buenos Aires, Av. Paseo Colón 850, Buenos Aires, Argentina.
E-mail: fsaccone@fi.uba.ar

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In this paper we describe the implementation of a biaxial vibrating sample magnetometer capable of performing measurements by the First Order Reversal Curve technique in an automated way. This magnetometer was employed for measuring the coefficient of anisotropy in an ALNICO piece. The measured value was $K_1 = (3.9\pm0.3) \times 10^5 J/m^3$. The interaction fields of this ALNICO sample have been analysed after performing its First Order Reversal Curves diagrams, in parallel and perpendicular to the field anisotropy orientation. For this last orientation, it was detected a low contribution of hysterons to the diagram in the near range of H=0 axis, as it can be expected due to a reduction in the remanence of perpendicular measurements of hysteresis loop.

Keywords: Biaxial Vibrating Sample Magnetometer (biaxial VSM); magnetic anisotropy; First Order Reversal Curves (FORC).

En este trabajo se describe la implementación de un magnetómetro de muestra vibrante biaxial con capacidad para realizar medidas de Curvas de Reversión de Primer Orden en forma automatizada. Este magnetómetro fue empleado para medir el coeficiente de anisotropía en una pieza de ALNICO. El valor medido fue $K_1 = (3.9\pm0.3) \times 10^5 J/m^3$. Los campos de interacción de esta muestra de ALNICO se han analizado a partir de los diagramas obtenidos midiendo de forma paralela y perpendicular a la orientación de la anisotropía de campo. Para esta última orientación, se detectó una baja contribución de histerones al diagrama en el rango próximo al eje H = 0, como es esperable debido a una reducción en la remanencia de los ciclos de histéresis medidos perpendicularmente.

Descriptores: Magnetómetro de muestra vibrante biaxial (VSM biaxial); anisotropía magnética; Curvas reversibles de primer orden (FORC).

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1. Introduction

There are different techniques that are useful to evaluate hysteresis cycles, from which it is possible to determine magnetic properties such as saturation, remanence, coercive field and maximum energy product. In particular, the most widely used are those that evaluate the electromotive force (emf) induced in sensor coils that collect changes in magnetic flux, resulting from the variation of the magnetisation of the sample with the applied field. Based in this principle, the vibrating sample magnetometers (VSM) became popular equipments in laboratories devoted to research in magnetic materials, since Foner [1] introduced its workability. In a VSM, the sample oscillates around an equilibrium position and this

movement induces the emf sensed by coils. Applied fields induce changes in magnetisation and in this way hysteresis loops are measured.

If a magnetic dipole oscilates in z direction, the emf induced in a set of sensor coils can be determined by means of:

$$\left(\frac{d\Phi}{dt}\right) = \sum_{i=1}^{N} \frac{dz}{dt} \vec{\nabla} B_i \cdot \vec{S}_i \tag{1}$$

where, Bi is the magnetic induction vector and Si is the area of the N turns that make up the coils. The above expression only is valid when B can be approximated by its value at the center of the coil. When the sample is small and its magnetisation is homogeneus, the so called reciprocity theorem [2] allows evaluating the flux in the coil (Φ) as:

$$\Phi = \mu_0 M \cdot H_n V - \frac{\partial \Phi}{\partial t} = -\mu_0 \vec{M} \cdot \frac{\partial \vec{H}_n}{\partial z} vV \tag{2}$$

being H_n the normalized sensor coil´s field evaluated at the sample location and V is the sample velocity as it oscillates in z direction.

The sensitivity along the direction of the applied field (here x) and perpendicular to it (here y) can be determined as:

$$S_x = \left(\frac{\partial H_{n,x}}{\partial z}\right), \qquad S_y = \left(\frac{\partial H_{n,y}}{\partial z}\right) \tag{3}$$

By means of a biaxial system it is possible to obtain the anisotropy constant as a direct measure. The energy of the system has two contributions: one anisotropy term EA and other, EP, that takes into account the interaction between M and the local field. For samples that have a strong uniaxial anisotropy the first term can be written as:

$$E_A = K_0 + K_1 \left(sin^2\theta\right) \tag{4}$$

where θ is the angle between M and the easy magnetisation axis of the sample. The other term corresponds to:

$$E_P = -\mu_0 H \cdot M = -\mu_0 M H \cos\left(\psi - \theta\right) \tag{5}$$

Where ψ is the angle between H and the easy magnetisation direction. At equilibrium, the minimum energy condition, $dE_T/d\theta = 0$, leads to:

$$K_1 \sin(2\theta) = \mu_0 M_y H \tag{6}$$

with $M_y = M\sin(\psi - \theta)$. By rotating the sample it is possible to find the angle for which M_y is maximum (easy axis) and the constant K_1.

The most efficient design to measure M parallel and perpendicular to H direction is a twelve-coil device [3,4]. If the sample oscillates along z direction and the applied field is parallel to x, then it is possible to measure M_X with the set of central coils while M_y can be determined using the lateral eight coils (see Bi3, B i4, B i5, B i6 coils shown at Fig. 1).

First order reversal curves (FORC) are partial hysteresis curves. The measurements begin by saturating the sample. Then each curve starts at some field value called reversal field (H_r) and proceeds back to positive saturation (see Fig. 2).

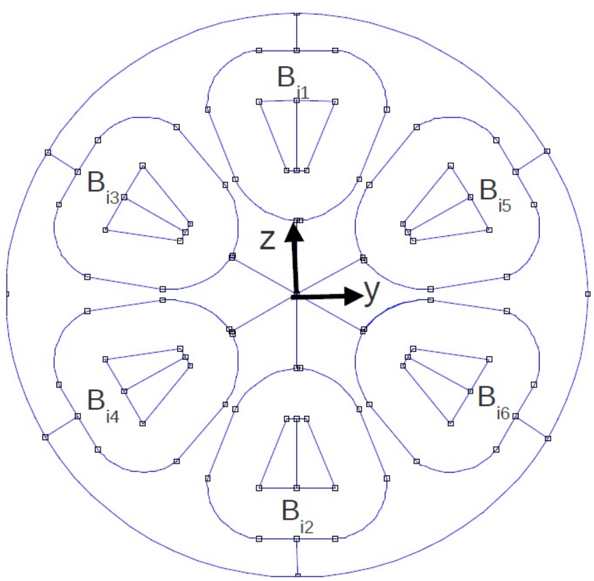

FIGURE 1. Schematic illustration of the arrangement of used pick-up coils.

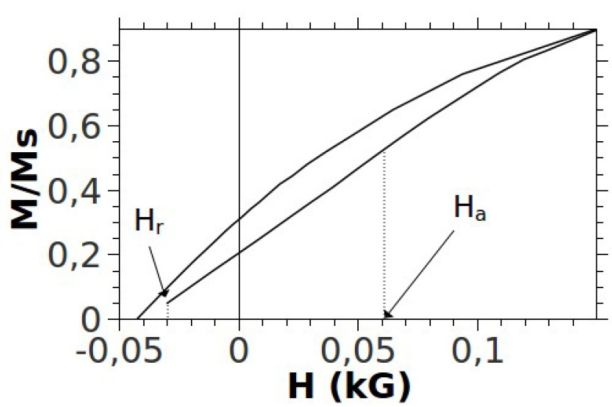

FIGURE 2. Schematic representation of a First Order Reversal Curve.

A set of FORCs helps us to obtain the FORC distribution defined as:

$$f(H_r, H_a) = \frac{-1}{2}\frac{\partial^2 M(H_r, H_a)}{\partial H_r \partial H_a} \tag{7}$$

where $M(H_r, H_a)$ is the magnetisation of the sample at applied field H_a on the FORC with reversal field H_r [5,6]. The FORC diagram method allows the characterisation of the switching field distribution as well as the interaction field distribution [7]. These diagrams provide information about the nature of the interactions and about the components of multiphase magnetic material. Thus, the FORC distribution becomes a powerful tool to describe the magnetic behavior of a system of particles [8]. Frequently, the more appropriate variables $h_c = (1/2)(H_r - H_a)$ and $h_u = (1/2)(H_r + H_a)$, are used (they correspond to a set of new axis oriented at 45^o with the original ones). The axis $h_c = 0$ stands for reversible interactions while $h_u = 0$ means that no bias is present.

2. Magnetometer description

As detection coils (pick-up coils) it was used a set of stator poles of a PC floppy drive $5(1/4'')$ that remained in the original assembly (scheme shown in Fig. 1). The six identical coils are located symmetrically on a circumference of 60 mm in diameter. The coils are connected in series, the top three as opposed to the bottom three. The set was fixed to the electromagnet pole pieces so that the coils plane is perpendicular to the field. The design allows a small gap (7mm) between pole pieces to increase the field and its uniformity. The electromagnet employed can hold up to 100V and has a maximum power of 1.3 kW. Power is supplied with a current source Kepco BOP 20 20M which can deliver up to 20A in continuous regime.

The device producing vibration of the sample consists of two 4” midrange audio speakers, assembled “back to back” with a coupling centering rod. This allows a passive compensation of mechanical vibration (resulting in a lowering of spurious signal components) and monitoring the signal by connecting an oscilloscope to one of the speaker. Then, the emf induced in the heart of the speaker responds as accurately as possible to the applied excitation.

To detect the magnetic flux collected by the coils a Lock-In amplifier (Perkin Elmer 7280 DSP) was used. The sensitivity of the coils was estimated by a numerical simulation. Free software, FEMM 4.0 [9], that allows solving magnetic problems with harmonic currents by the finite element method was used. The boundary conditions used were: $A_\tau = 1Wb/m$ on both edges of the turns and $\partial A_n/\partial r = 0$ on the radial edges. Since the coils were connected in series and opposition the phase shift between excitacions was chosen asp π.

With these simulations, we determined the sensitivity to displacements in the y and z directions (see Fig. 3).

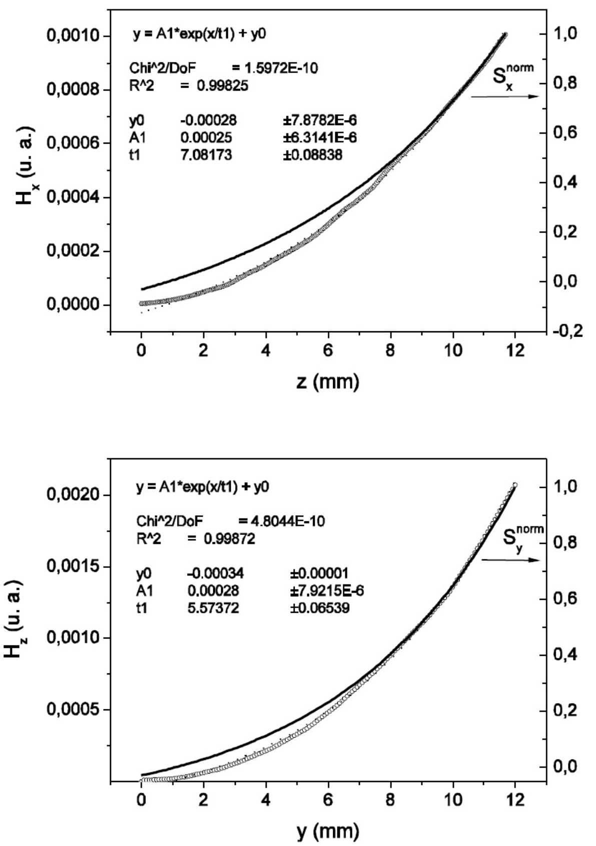

FIGURE 3. Sensitivity in y and z directions.

In order to detect the sensitivity range of our system we measured the hysteresis loop of an amorphous ribbon, used as transformer core, in parallel-field configuration, with an electromagnet gap of 32 mm. The measurement was made by centering the vibrating rod at the position that was determined as the most sensitive by the FEM calculus. The sample weights around 2 mg. As it can be observed from Fig. 4, the SNR is appropriated and we estimated from this loop a sensitivity for our VSM of around 1 emu/g.

3. Results

3.1. Anisotropy

An ALNICO magnet (VIRASON S. A.) with a mass of 0.9391 g was fixed to the vibrating rod in a central position of the electromagnet gap. The vibrator system was mounted on a goniometer. The outer pick-up coils, connected as indicated in Bernards' reference [3], sense

the emf produced by oscillations of the transversal magnetisation M_y vector. In that work, the author selects the positive polarity of coils so that their flux increase when north (or south) pole of the magnetic dipole approaches (or moves away) from them. In Fig. 5, the transversal magnetisation (M_y) as a function of sample orientation is shown. The M_y vector follows the sine function described in Eq. 6. From fitting of the amplitude of the experimental curve and using that equation, the uniaxial anisotropy K_1 for the ALNICO piece was determined, using a demagnetizing coefficient (or shape factor) N of 0.05, with a value of $(3.9 \pm 0.3) \times 10^5 J/m^3$.

3.2. First Order Reversal Curves (FORC)

The same sample employed for anisotropy determination, was used in order to make the corresponding FORC diagrams that are shown in Fig. 6a and 6b. A set of fifty FORC curves was measured for two different orientations of the major axis of the sample, parallel and perpendicular to the applied field. The diagrams were calculated with a routine of finite differences that approximated derivatives in Eq. (7). Two distinctive characteristics are visible when comparing both diagrams: First, the bright zone in the diagonal of Fig. 6b, corresponds to reversible hysterons contribution $(h_c = 0 axis)$ that are absent when the sample is measured with its axis perpendicular to H_a . This fact is expected for the hysteresis loop, since samples measured in perpendicular directions to their easy axis, are rotated along a 45° direction in the M-H plane (with a lower contribution to magnetization in the center of the loop). Second, the darkest zone in both diagrams corresponds to maxima in the hysteron population distribution. As it can be observed, for parallel-to-field axis direction (see Fig. 6 a) this maximum is located around 0.5 kG. In the other case, the maximum is around 1 kG (see Fig. 6b) coincident with a higher coercive field detected for perpendicular orientation, because magnetisation is forced to move out 90° from the easy axis.

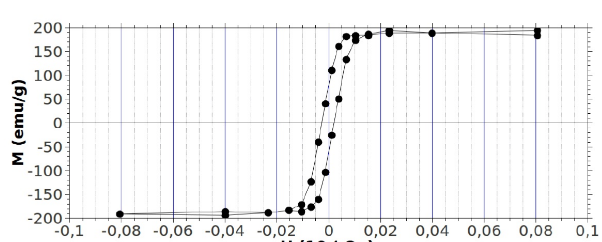

FIGURE 4. Amorphous ribbon hysteresis loop measured to estimate the VSM sensitivity.

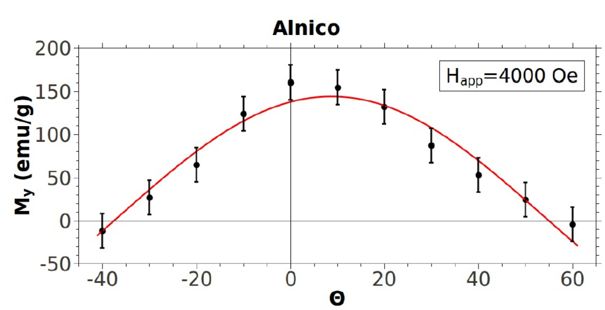

FIGURE 5. Uniaxial anisotropy determination of an ALNICO magnet. The applied field at saturation was 4000 Oe.

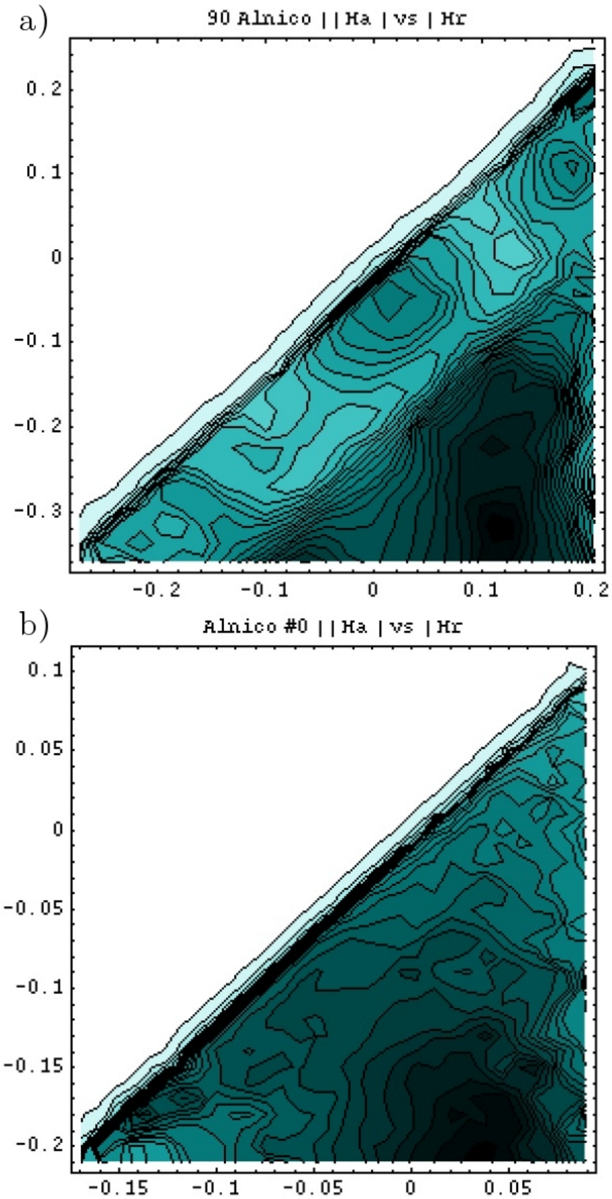

FIGURE 6. Alnico FORC diagrams measured for two different orientations of the major axis of the sample: a) parallel; b) perpendicular to the applied field.

4. Conclusions

In this work, we show the technical characteristics of a homemade vibrating sample magnetometer. For its set up, we used a Lock In amplifier and a programmable courrent source, managed by a PC through a GPIB comunication port. Our VSM was designed also to perform perpendicular-to-field magnetisation measurements. As sensing coils we used a set of coils that corresponds to part of an unused $51/4''$ diskette driver. In order to estimate the position dependence of sensitivity, simulations by finite element models were made. This parameter was determined as 1 emu/g. In measurement of the perpendicular component of the magnetisation, it is possible to obtain the magnetic anisotropy of the sample with the use of a goniometer. In the case of a piece of Alnico we determined an anisotropy constant consistent with the value found in bibliography [10].

In addition, we automated the FORC curve measurements with a program written in G language. A set of 50 partial hysteresis curves was required for a good magnetic characterisation by FORC diagrams. Perpendicular-to-field FORC diagram, exhibited a low contribution of reversible interactions and a magnetic hardening, respect to longitudinal FORC diagrams.

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