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Criteria for saturated magnetization loop

A. Harres [a], M. Mikhov [b], V. Skumryev [c,d], A.M.H. de Andrade [e], J.E. Schmidt [e], J. Geshev [d,e,*]

[a] Departamento de Física, UFSM, Santa Maria, 97105-900 Rio Grande do Sul, Brazil
[b] Faculty of Physics, University of Sofia, 1164 Sofia, Bulgaria
[c] Institució Catalana de Recerca i Estudis Avançats, 08010 Barcelona, Spain
[d] Departament de Física, Universitat Autònoma de Barcelona, 08193 Barcelona, Spain
[e] Instituto de Física, UFRGS, Porto Alegre, 91501-970 Rio Grande do Sul, Brazil

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ABSTRACT

Proper estimation of magnetization curve parameters is vital in studying magnetic systems. In the present article, criteria for discrimination non-saturated (minor) from saturated (major) hysteresis loops are proposed. These employ the analysis of (i) derivatives of both ascending and descending branches of the loop, (ii) remanent magnetization curves, and (iii) thermomagnetic curves. Computational simulations are used in order to demonstrate their validity. Examples illustrating the applicability of these criteria to well-known real systems, namely Fe_3O_4 and Ni fine particles, are provided. We demonstrate that the anisotropy-field value estimated from a visual examination of an only apparently major hysteresis loop could be more than two times lower than the real one.

1. Introduction

The observation of hysteresis in magnetization (M) versus applied magnetic field (H) variations is a fingerprint of irreversible magnetization processes. These hysteresis loops are employed to characterize magnetic materials based on parameters like the coercive field (H_C) and remnant magnetization (M_R). Because of hysteresis, there is an infinite number of different ways to reach a given $M(H)$ state. However, when a high enough field is applied, M becomes a single-valued function of H since the pre-existing domain configuration is wiped out by the field (phenomena like spin-flopping, that can take place in antiferromagnetic and other multi-sublattice structures, will not be considered here). The minimum field value at which a single-valued $M(H)$ is reached is the anisotropy field H_A (sometimes, the term 'closure field' is also used) which, in general, depends on the intrinsic temperature-dependent anisotropy constants and saturation magnetization (M_S) of the material. The classical concept of magnetic saturation implies M which does not change with H at 0 K. However, if M varies mostly through rotation before saturation, this concept only holds for single-crystal materials when **H** is applied along an easy- or a hard-magnetization axis; for all other orientations, $M(H)$ is a reversible function for $|H| > H_A$, with $M = M_S$ being only at $|H| = \infty$ [2] (at finite temperatures there is an additional contribution to the slope of $M(H)$ due to paramagnetism, i.e., the tendency of the magnetic moments to align with H). This is what we further refer to as saturated (or effectively saturated) state.

A magnetization hysteresis loop is traced when H is cycled between two extreme-field values, with H^+_{max} and H^-_{max} being, normally, H^+_{max} positive and H^-_{max} negative. When the values of both H^+_{max} and $|H^-_{max}|$ are higher than H_A, the curve is called major loop. Those measured under any other conditions are known as minor loops [1], which may present a variety of shapes, depending on H^+_{max} and H^-_{max}, on the magnetic state history and on the system being studied. The number of times the field is cycled may also play an important role [3]. Often, minor loops do not close on themselves on the first cycle. Different phenomena are associated with this effect [4]. The magnetic aftereffect describes the evolution of the magnetization with time for a fixed magnetic field. Accommodation (or reptation) effect accounts for a drift of a minor loop toward an equilibrium one when H oscillates within a given interval [5]. Finally, the effect of interaction fields attaining values during a minor loop that were not reached previously is referred to as magnetic flip-flop effect [6].

Arguably, minor loops contain more information about the magnetic state than major ones. Series of curves traced for increasing $|H^-_{max}|$ and/or H^+_{max} are used to study the magnetization processes; in principle, it is possible to differentiate between nucleation-dominated and wall-pinning-dominated reversals using this technique [7]. The first-order reversal curve (FORC) diagrams, which are a powerful method to study magnetization reversal

* Corresponding author at: Instituto de Física, UFRGS, Porto Alegre, 91501-970 Rio Grande do Sul, Brazil.
E-mail address: julian@if.ufrgs.br (J. Geshev).

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mechanisms and quantify magnetic interactions, are based on transformation of minor-hysteresis-loop data [8].

Asymmetry between ascending and descending branches of $M(H)$ curves, along with their horizontal and vertical displacements, are common features of minor loops. Such behavior may be misleading, and sometimes it is wrongly associated with exchange bias (EB) [9], which comes from the magnetic exchange coupling between a ferromagnet (FM) and uncompensated spins at the interfacial region of an adjacent antiferromagnet (AF). The shift of the magnetization curve along the field axis is the most known manifestation of the effect, vastly applied in magnetoelectronic devices and still intensively explored. With the advent of production technologies and the development of powerful experimental techniques, complex magnetic systems are currently under investigation. During the last two decades, a great number of articles have been published that report $M(H)$ curves shifted along the H-axis and, in many cases, also along the M-axis. In many of these works, however, the magnetization does not seem to be saturated in at least one of the branches of the hysteresis cycle, thus leading to possible incorrect interpretations of the observed shifts.

Determining whether a hysteresis loop is a minor or a saturated one is a problem faced in a daily basis by experimentalists studying magnetic systems. Usually, it is believed that a simple visual inspection of the high (positive and negative) field regions of a hysteresis loop is sufficient to verify whether unambiguous reversible rotation is reached or not. Overlapping of certain number of data from the high-field end of the ascending/descending branches of the loop or merging the two branches is often presumed to guarantee saturated loop. In many cases, though, such visual judgment can be misleading.

The aim of this paper is to draw attention to this problem and to present some objective criteria to determine whether a hysteresis loop is actually a major one. We hope that it could be of interest not only for researchers peripheral to magnetism but also for those actively working with magnetic materials and structures. Because of the particularly important consequences of the type of the hysteresis loop on the EB systems we will pay a special attention to this case. Last but not least, we believe that the article could help slowing down the growing rate of papers containing inaccuracies steaming from not discriminating between minor and major loops. Since criticizing specific publications is not our intention, we avoided citing them.

2. Minor loops and exchange bias

The mere existence of a FM/AF interface in a system does not guarantee that it will present EB. The effect is initialized either by applying magnetic field during the samples' production or cooling down the sample through the AF's Néel temperature [9] or by post-deposition ion bombardment [10,11], or even by applying sufficiently large fields at a fixed temperature [12]. In some of the aforementioned systems, experimental results suggest the existence of the so-called spontaneous exchange bias (SEB) [13], where the first field applied during a hysteresis loop trace induces an EB axis; unfortunately, such conclusions are not rarely withdrawn from unsaturated curves. This does not mean that EB (or SEB) cannot be observed in these structures; however, each case should be carefully analyzed since if the data presented to infer the existence of the effect are not properly obtained, the minor loop's effects cannot be completely ruled out.

Recall that reliable parameters, including the EB ones, are extracted from hysteresis loops that show reversible parts, i.e., high (both positive and negative) field regions where the outward and return parts of $M(H)$ coincide for large enough range of fields [14].

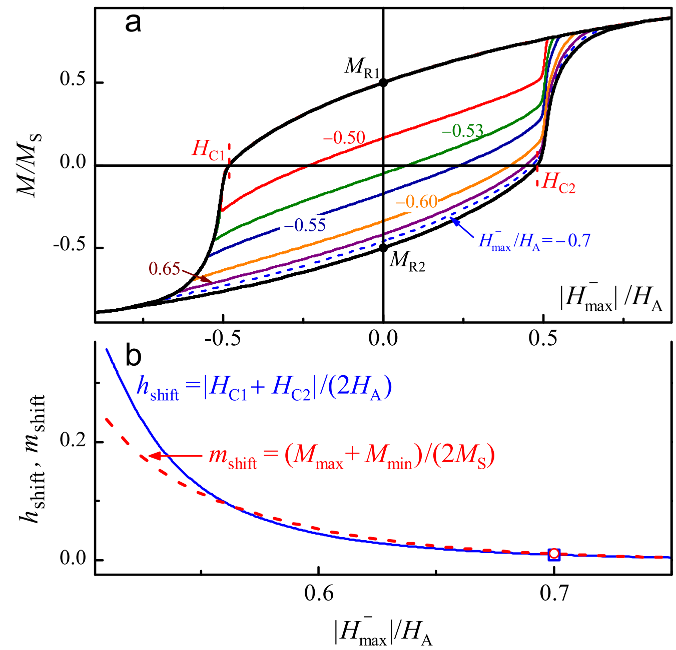

Fig. 1. (a) A major hysteresis loop and a series of minor ones for a disordered system of non-interacting single-domain uniaxial-anisotropy particles, calculated for $|H^-_{max}|/H_A$ ratios higher than 0.5 and for starting field (H^+_{max}) higher than H_A; the curve enveloping all others is the major loop. The sufficient-for-saturation value of H^+_{max} results in descending branches that coincide with the major one for all minor loops. Although H^+_{max}/H_A of 1.2 is used for all curves, only the $(-0.9, +0.9)$ H/H_A region is shown for better visualization. (b) The shifts along the field ($h_{shift} = \frac{1}{2}|H_{C1} + H_{C2}|/H_A$) and magnetization [$m_{shift} = \frac{1}{2}(M_{max} + M_{min})/M_S$, where M_{max} and M_{min} are the magnetization values at H^+_{max} and H^-_{max}, respectively] axes versus $|H^-_{max}|/H_A$ extracted from these minor loops, where the symbols correspond to $H^-_{max}/H_A = -0.7$.

A correct determination of the magnetization curve characteristics is crucial for the discussions presented below; detailed definitions of the key parameters of a hysteresis loop can be found in a recent work [15]. We recall that, usually in exchange-coupled systems, the absolute value of coercivity of the descending branch of a hysteresis loop, H_{C1} (i.e., the value of H at which $M=0$), does not have the same value as that of the ascending branch, H_{C2}, and $H_C = \frac{1}{2}(H_{C2} - H_{C1})$; the respective remnant magnetizations of the descending and ascending branches of a major hysteresis loop, M_{R1} and M_{R2}, could differ greatly in value and may even have one and the same sign. The parameters of a major hysteresis loop H_{C1}, H_{C2}, M_{R1} and M_{R2} are visualized in Fig. 1(a).

In their extensive review on EB in nanostructures, Nogués et al. [16] stressed that minor loops may exhibit shifts characteristic of all unsaturated FM materials that have no relation to EB. Although several comments on this problem have also been published [17–19], the rate of publications, where the attainment of magnetization's reversibility of presumably shifted curves has not been verified, keeps increasing. In some reports, unlike the conventional EB, the shifts strongly depend on H^-_{max} and are totally removed if this is high enough, confirming that these shifts are due to unsaturated samples.

The authors of Ref. [20], being aware of the minor-loop problem in EB systems, have tried to establish a criterion whether a hysteresis loop is a major one or not. They examined the magnetizations M_{max} and M_{min} at H^+_{max} and H^-_{max}, respectively, and the value of the shift along the M-axis defined as $m_{shift} = \frac{1}{2}(M_{max} + M_{min})/M_S$; it has been assumed that if the value of m_{shift} becomes of the order of the error margin of the measurement technique, then the corresponding magnetization curve has effectively attained saturation and, if it is shifted along the H-axis