

WHITE PAPER

First-order reversal curves enhance understanding of nanoscale magnetic materials

LAKE SHORE CRYOTRONICS, INC. B.C. Dodrill and L. Spinu

Magnetic nanovatices, nanoparticles make up an imporagnetic nanowires, nanodots, and tant class of nanostructured magnetic materials. Due to size confinement in the nanometer range, new phenomena arise in these materials. These structures are ideal candidates for technological applications in spintronics, high-density recording media, microwave electronics, and permanent magnets as well as for medical diagnostics and targeted drug delivery applications. In addition to being of technological value, these materials represent an experimental playground for fundamental studies of magnetic interactions and magnetization mechanisms at the nanoscale level.^{1,2}

When investigating the magnetic interactions in these materials, one of the most interesting configurations is a periodic array of magnetic nanowires because both the size of the wires and their arrangement with respect to one another can be controlled. Inter-wire coupling is one of the most important effects in nanowire arrays because it significantly affects magnetization switching as well as microwave and magneto-transport properties. Experimentally, one of the most widely used methods to investigate the strength and measure the effects of these interactions is by using a magnetometry technique that measures and analyzes first-order reversal curves (FORCs).3

A FORC is measured by saturating a sample in a field H_{sat} , decreasing the field to a reversal field H_a , then sweeping the field back to H_{sat} in a series of regular

field steps H_b. This process is repeated for many values of H_a yielding a series of FORCs. The measured magnetization at each step as a function of H_a and H_b gives M(H_a,H_b) which is then plotted as a function of H_a and H_b in field space. The FORC distribution ρ (H_a, H_b) is the mixed second derivative, that is, ρ (H_a,H_b) = $-\partial^2 M(H_a,H_b)/\partial H_a \partial H_b$, and a FORC diagram is a contour plot of ρ (H_a,H_b) with the axis rotated by changing coordinates from (H_a,H_b) to H_c=(H_b-H_a)/2 and H_u=(H_b+H_a)/2 where H_u corresponds to the distribution of interaction fields, and H_c the distribution of switching fields.

The magnetic response of a material is proportional to its intrinsic magnetism and the volume of material being measured. For nanoscale magnetic materials, the volume is inherently small and consequently the signal that is measured by a magnetometer is very low. Thus, the sensitivity of the magnetometry technique is an important parameter in connection with characterizing nanostructured magnetic materials.

Magnetometry techniques can be broadly classified into two categories: inductive and force-based. Common inductive methods include vibrating sample magnetometry (VSM), extraction magnetometry, AC susceptometry, and superconducting quantum interference device (SQUID) magnetometry. The two most commonly used inductive techniques are VSM and SQUID magnetometry. Alternating gradient magnetometry (AGM) is the most often used force-based technique.

Vibrating sample magnetometry

In vibrating sample magnetometry, originally developed by Simon Foner⁴ of MIT's Lincoln Laboratory, a magnetic material is vibrated within a uniform magnetic field H, inducing an electric current in suitably placed sensing coils. The resulting voltage induced in the sensing coils is proportional to the magnetic moment of the sample. The magnetic field may be generated by an electromagnet or a superconducting magnet. VSM measurements can be performed from <4.2 K to 1273 K using integrated cryostats or furnaces.

Commercial VSM systems provide measurements to field strengths of ~ 3 T (30,000 Oe) using conventional electromagnets, as well as systems employing superconducting magnets to produce fields to 16 T. When used with electromagnets, very small step changes in field can be made (i.e., ~ 1 mOe) and the measurement is very fast. A typical hysteresis loop measurement can take as little as a few seconds to a few minutes.

When used with superconducting magnets, higher field strengths are possible; however, this limits the field setting resolution, and the measurement speed is inherently slower due to the speed at which the magnetic field can be varied using superconducting magnets. Additionally, magnetometers employing superconducting magnets are more costly to opperate since they require liquid helium. However, they reach higher magnetic fields than air- or water-cooled electromagnets, which is necessary to saturate some magnetic materials, such as rareearth permanent magnetic materials.

The noise floor of commercially available VSMs is 10⁻⁷ emu. This high sensitivity is sufficient for many magnetic materials. However, for materials that are magnetically diluted, or materials that possess a very small sample volume, as is the case with nanoscale magnets, this sensitivity may be insufficient to properly characterize such materials.

SQUID magnetometry

Quantum mechanical effects in conjunction with superconducting detection

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B.C. Dodrill, Lake Shore Cryotronics, Inc.

L. Spinu, Advanced Materials Research Institute, University of New Orleans

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coil circuitry are used in SQUID-based magnetometers to measure the magnetic properties of materials. Theoretically, SQUIDs are capable of achieving sensitivities of 10⁻¹² emu, but practically, they are limited to sensitivities of 10⁻⁸ emu because the SQUID also picks up environmental noise. As in a VSM, SQUIDs may be used to perform measurements from low to high temperatures (from <2 K to 1000 K). Superconducting magnets with field strengths up to 7 T are employed in SQUIDs5,6; therefore, the measurement is inherently slow due to the speed at which the magnetic field can be varied, as is the case for superconducting magnet-based VSM systems. A typical hysteresis M(H) loop measurement can take one hour or more. This also means that FORC measurements are impractical when using a SQUID magnetometer. The FORC measurements require the acquisition of thousands of data points, which would take several days using a SQUID magnetometer and be very expensive due to the cost of liquid helium.

Alternating gradient magnetometer

Force methods involve determination of the apparent change in weight for a material when placed in an inhomogeneous magnetic field. The sample experiences a force *f* along the axis of the field gradient (dH/dz), which is given by f = m(dH/dz) where *m* is the magnetic moment. The equipment required for such force methods are either an electro- or superconducting magnet, and a balance for force measurements.

A commercial variant of these methods is the alternating gradient magnetometer. AGMs are capable of achieving sensitivities in the 10^{-8} to 10^{-9} emu range, and like the VSM, the AGM is a very fast measurement; a typical hysteresis loop takes seconds to minutes. Commercial AGM systems can be used for ambient and low-temperature measurements (~10–300 K) to the moderate 2–3 T fields achievable with electromagnets.

Figure 1 shows a series of FORCs measured using an AGM for a periodic array of Ni nanowires with a mean diameter of 70 nm and an inter-wire distance



Figure 1. First-order reversal curves (FORCs) for an array of magnetic Ni nanowires. Data were recorded at Lake Shore using an AGM; the sample was fabricated by L. Spinu at UNO.

Figure 2. Distribution of interaction fields as determined from FORC analysis.⁷

of 250 nm. The nanowire samples were fabricated by electrodeposition using an anodic aluminum oxide membrane as a template. As an example of AGM measurement speed, the FORCs consist of 4640 points and the data were recorded in only 20 minutes. Analysis of these FORCs yields the local interaction H_u and coercive H_c fields distributions shown in Figure 2. This measurement protocol and analysis provide information regarding irreversible magnetic interactions or processes in this array of nanoscale wires that cannot be obtained from the standard hysteresis loop measurement.

Magnetic measurements are indispensable in characterizing the magnetic properties of nanoscale magnetic materials. This review briefly described the more common inductive and force-based magnetometry techniques currently used to characterize their magnetic properties. FORC measurements provide a means for understanding nanoscale magnetic interactions in these materials, which is requisite to their eventual use in commercial products. Materials research such as this is key to innovation in numerous consumer products, including computers, communications equipment, and electrical motors.

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