First-Order-Reversal-Curve (FORC) Measurements of Nano-Magnetic Materials

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Abstract

The magnetic characterization of nanoscale materials is usually made by measuring a hysteresis loop. It is not possible to obtain interaction or coercivity distribution information from the hysteresis loop alone, however. First-order-reversal-curves (FORCs) provide insight into the relative proportions of reversible and irreversible components of the magnetization of a material. This paper will discuss the FORC measurement technique and subsequent analysis which lead to the FORC diagram, and present examples of measurement results for different magnetic materials.

About Lake Shore Cryotronics, Inc.

Supporting advanced research since 1968, Lake Shore (http://www.lakeshore.com) is a leading innovator in measurement and control solutions for materials characterization under extreme temperature and magnetic field conditions. High-performance product solutions from Lake Shore include cryogenic temperature sensors and instrumentation, magnetic test and measurement instruments, probe stations, and precision materials characterizations systems that explore the electronic and magnetic properties of next-generation materials. Lake Shore serves an international base of research customers at leading university, government, aerospace, and commercial research institutions and is supported by a global network of sales and service facilities.

First-Order-Reversal-Curves (FORCs) Improve Insight

Magnetometric measurements can reveal important characteristics and behaviors of nanoscale magnetic materials such as thin films, composites, dots, wires, and arrays. The measurement most commonly performed to characterize a material's magnetic properties is that of a major hysteresis loop. As researchers seek information beyond the scope of hysteresis loop measurements such as interaction or coercivity distribution, another technique becomes necessary. First-order-reversal-curve (FORC) measurements can provide insight into the relative proportions and irreversible components of the magnetization of various materials. Nanoscale magnetic materials to which FORCs can be applied include:

- Exchange-coupled nanocomposite permanent magnet materials for which FORC enables differentiation of the magnetically hard and soft phases[1].
- Arrays of magnetic nanowires, nanodots or nanoparticles for which FORC enables investigation of irreversible magnetic interactions or processes in the array due to coupling between adjacent wires, dots or particles[2].
- Exchange-biased magnetic multilayer **thin films** for which FORC enables the correlation of exchange bias with in-homogeneities existing at the antiferromagnetic/ferromagnetic interface[3].

Magnetization Measurements and FORCs

The hysteresis or M(H) loop is typically used to determine a material's saturation magnetization M_s (the magnetization at maximum applied field), remanence M_r (the magnetization at zero applied field after applying a saturating field), and coercivity H_c (the field required to demagnetize the material). More complex magnetization curves covering states with field and magnetization values located inside the major hysteresis loop, such as FORCs, can provide additional information that can be used to characterize magnetic interactions.

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 $\rho(H_a, H_b)$ is the mixed second derivative, i.e., $\rho(H_a, H_b) = -\partial^2 M(H_a, H_b)/\partial H_a \partial H_b$, and a FORC diagram is a contour plot of $\rho(H_a, H_b)$ 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 represents the distribution of interaction fields, and H_c represents the distribution of switching fields.

FORCs can be measured by using any type of magnetometer that measures the DC or static magnetic properties of a material. The most common techniques are vibrating sample magnetometry (VSM), superconducting quantum interference device magnetometry (SQUID) and alternating gradient magnetometry (AGM). VSMs are commonly used because measurements can be performed for a broad range of material magnetizations (10^{-6} to > 10^3 emu) and because measurements can be performed over a broad range of temperatures (4 K to 1,273 K) and magnetic fields, employing either electromagnets (3 T)[4,5] or high-field superconducting magnets (16 T)[6,7]. The primary advantage of SQUID[6,7] and AGM[4] systems are their sensitivity, which are 10^{-8} and 10^{-9} emu, respectively. They are most commonly used to measure very weak magnetic samples. Since a typical sequence of FORCs may contain thousands of data points, measurement speed is very important. It is most convenient to use electromagnet-based magnetometers for FORC measurements because the speed with which the magnetic field can be varied is considerably faster than is the case for superconducting magnet-based systems.

Typical Magnetic Measurement Results

FORC measurement and analysis were performed for a nanocomposite hybrid soft/hard permanent magnet, an ordered array of nickel nanowires, and an exchange bias magnetic multilayer thin film.

Permanent Magnets

Rare-earth permanent magnet materials are indispensable elements in many electronic devices such as electrical motors, hybrid vehicles, and portable communications devices. The magnets have major influence on the size, efficiency, stability and cost of these devices and systems. Over the last couple of decades there has been interest in the development of nanostructured magnets and exchange-coupled nanocomposite alloys with co-existing soft and hard phases because of the coercivity enhancement that is obtained at the single-domain size (nanometer scale).

Figure 1 shows a VSM[4] major hysteresis loop measurement for a nanocomposite magnet containing a mixture of magnetically soft (ferrite) and hard (SmCo) phases. From the hysteresis loop measurements of the samples separately, the coercivities were 275 Oe (ferrite) and 900 Oe (SmCo). The hysteresis loop for the mixed sample shown in figure 1 is clearly dominated by the hard (SmCo) phase (H_c = 900 Oe). It is not possible to differentiate between the soft and hard phases from the hysteresis loop alone.

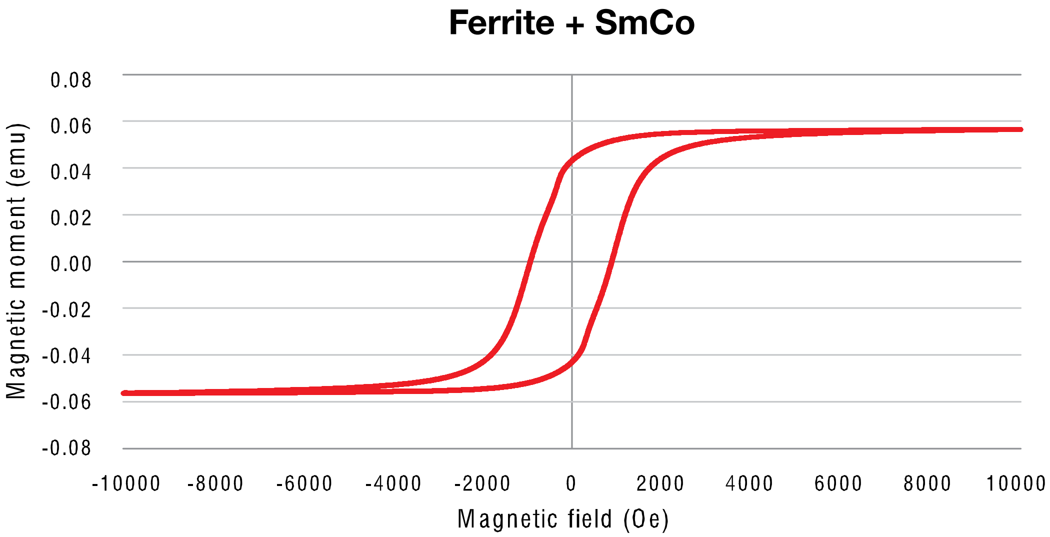

Figure 1. Major hysteresis loop for a mixture of ferrite and SmCo nanopowders

Figures 2 and 3 show a series of VSM[4] FORCs and the FORC diagram[8] for the mixed sample. For clarity, contours have been added to the FORC diagram. The soft (ferrite) and hard (SmCo) phases are clearly differentiated, demonstrating the utility of FORC analysis for characterizing the magnetic properties of magnets containing both soft and hard phases.

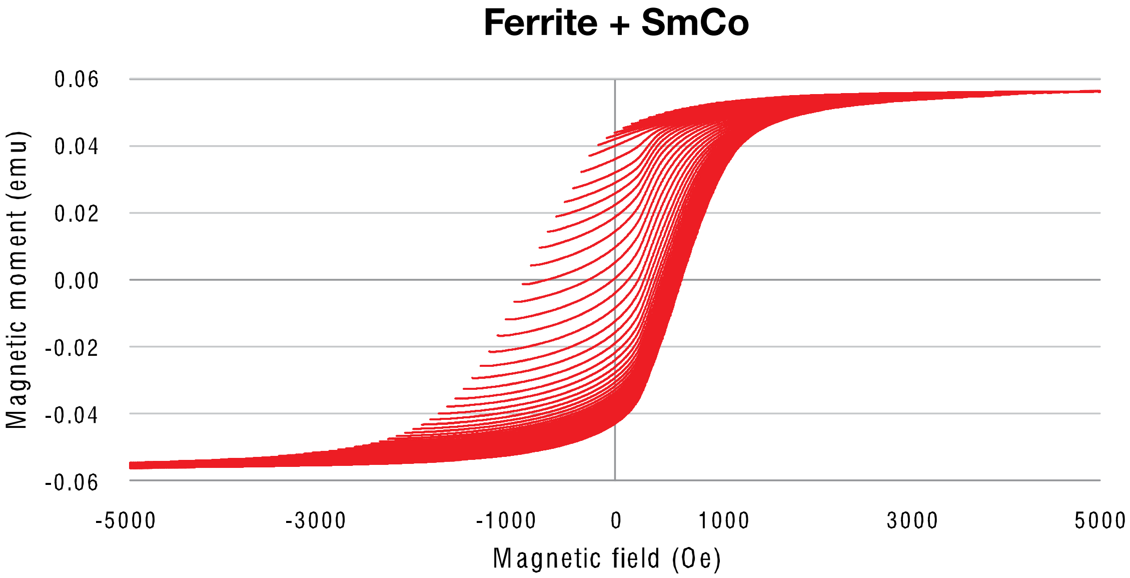

Figure 2. FORCs for a mixed ferrite (soft) and SmCo (hard) sample

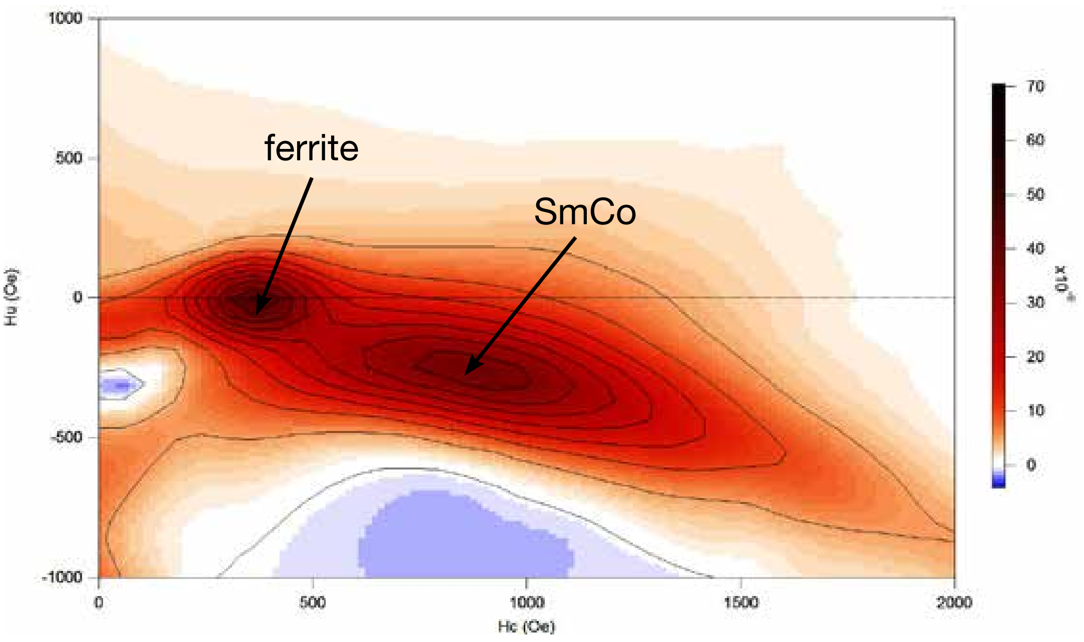

Figure 3. FORC diagram with contours for a mixed ferrite (soft) and SmCo (hard) sample

Magnetic Nanowire Arrays

Magnetic nanowires, nanodots and nanoparticles are an important class of nanostructured magnetic materials. At least one of the dimensions of these structures is in the nanometer (nm) range and thus, new phenomena arise in these materials due to size confinement. These structures are ideal candidates for important technological applications in spintronics, high density recording media, microwave electronics, permanent magnets, and for medical diagnostics and targeted drug delivery applications. In addition to technological applications, these materials represent an experimental playground for fundamental studies of magnetic interactions and magnetization mechanisms at the nanoscale level. 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 and microwave and magneto-transport properties. Experimentally, FORCs are used to investigate the effect and strength of these interactions.

Figure 4 shows a series of FORCs measured using an AGM[4] for a periodic array of Ni nanowires with a mean diameter of 70 nm and an inter-pore distance of 250 nm[9]. The nanowire samples were fabricated by electro-deposition using anodic aluminum oxide membrane as a template. As an example of AGM measurement speed and sensitivity, the FORC curves consist of 4,640 points, and the data was recorded in only 20 minutes despite the weak magnetization (450 μemu). Analysis[8] of these FORC curves yields the local interaction H_u and coercive H_c field distributions shown in Figure 5. This measurement protocol and analysis provide additional information regarding irreversible magnetic interactions or processes in this array of nanoscale wires, which cannot be obtained from the standard hysteresis loop measurement.

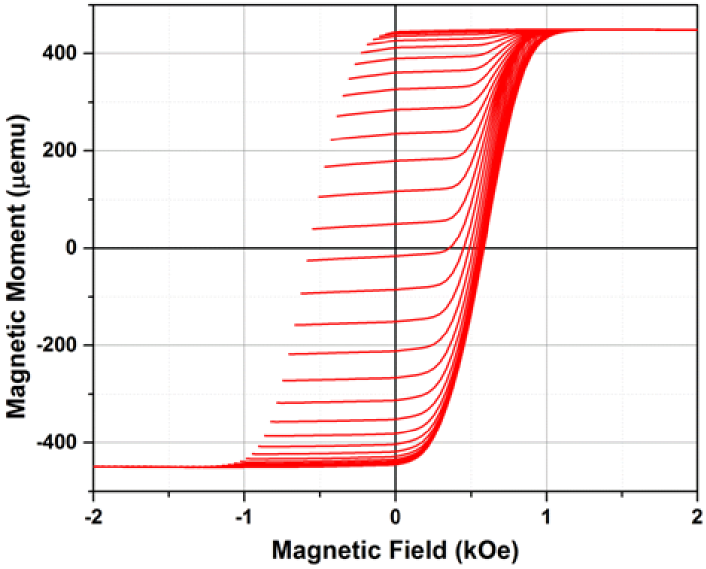

Figure 4. First-order-reversal-curves (FORCs) for an array of magnetic nanowires

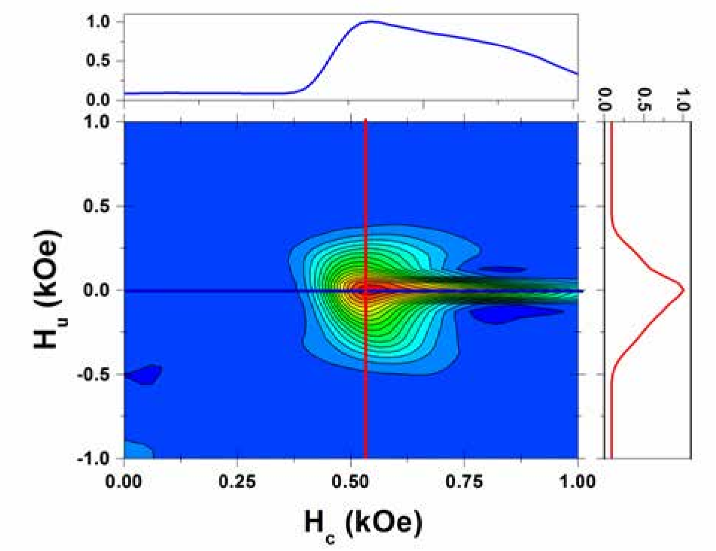

Figure 5: Distribution of interaction and coercivity fields as determined from FORC analysis

Exchange Bias Magnetic Multilayer Films

Exchange bias magnetic multilayer films are technologically important materials for applications such as spin-valve read heads for hard disk drives and gigahertz-range microwave devices. In these materials at least one antiferromagnetic (AFM) layer is intercalated between ferromagnetic (FM) layers. In addition to their technological applications, they are also useful for fundamental studies of magnetic interactions and magnetization reversal processes in magnetic nanostructures because both the number (n) of periodic arrays and the thickness of the FM and AFM layers can be controlled.

Figure 6 shows VSM[4] hysteresis loops with the applied field oriented in-plane, and either parallel to the easy axis (0° and 180°) or perpendicular to the easy axis (90°) for a multilayer film[10] of composition [FeNi (60 nm)/IrMn (20 nm)]$_n$, where FeNi represents Ni (80%) Fe (20%), and the number of layers n = 5.

When the magnetic field is applied parallel (0°) to the exchange bias field the loop is shifted towards the left (negative field values), and when applied anti-parallel (180°) the loop is shifted to the right (positive field values). For magnetic fields oriented perpendicular to the exchange bias field (90°), the loop passes through the origin and has zero coercivity ($H_c = 0$). From the 180° hysteresis loop the exchange bias and coercivity fields are: $H_{ex} = 40$ Oe and $H_c = 3.5$ Oe. The extra steps in the 0° and 180° curves between positive and negative saturation magnetization are related to microstructural defects/roughness of the AFM/FM interfaces. FORC analysis can give a more detailed account of the effect of in-homogeneities on the magnetization reversal of the AFM/FM interface.

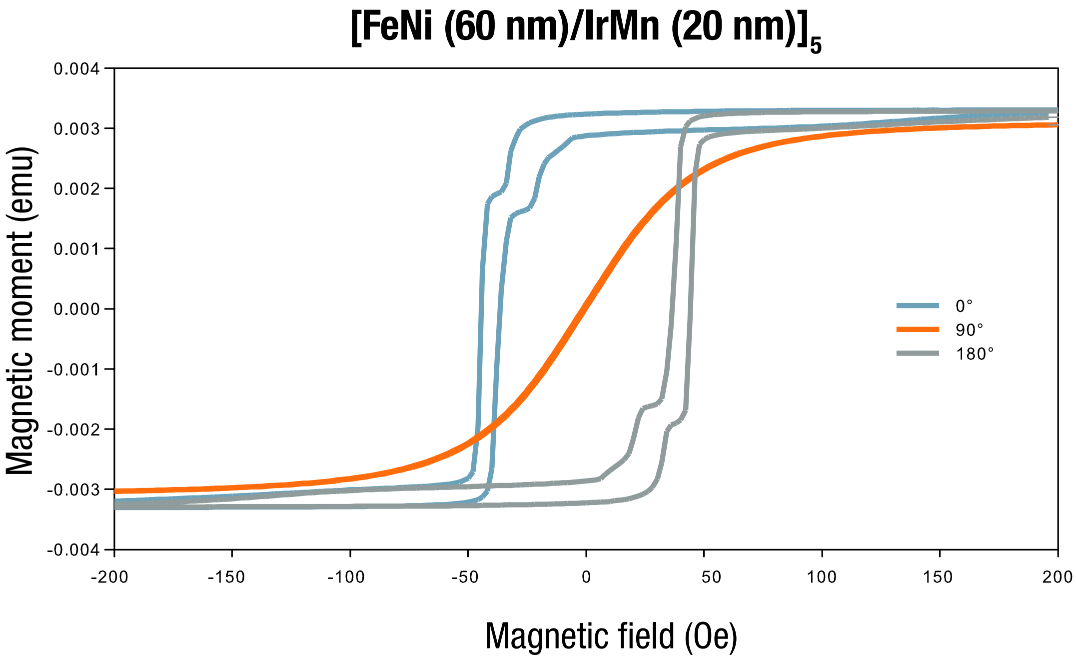

Figure 6. Hysteresis loop for three different in-plane orientations (0°, 90° and 180°)

Figure 7 shows a series of VSM[4] FORCs for the field oriented at 180° with respect to the exchange bias field, and figure 8 shows the corresponding FORC diagram[8]. The FORC diagram in figure 8 reveals a main FORC distribution that is centered around H_c (3.5 Oe); however, the distribution of switching fields extends over several Oe. The peak of the distribution in the H_u direction corresponds to the exchange bias field H_{ex} (40 Oe). The spread of the distribution in the H_u direction is related to interactions between the AFM and FM layers. The satellite distribution centered at H_u = 25 Oe and H_c = 6 Oe is related to structural inhomogeneities at the AFM/FM interface and are more pronounced the higher the number of layer repetitions, or equivalently the higher the number of AFM/FM interface in-homogeneities. The FORC measurement protocol and analysis provide additional information that cannot be obtained from the standard hysteresis loop measurement alone.

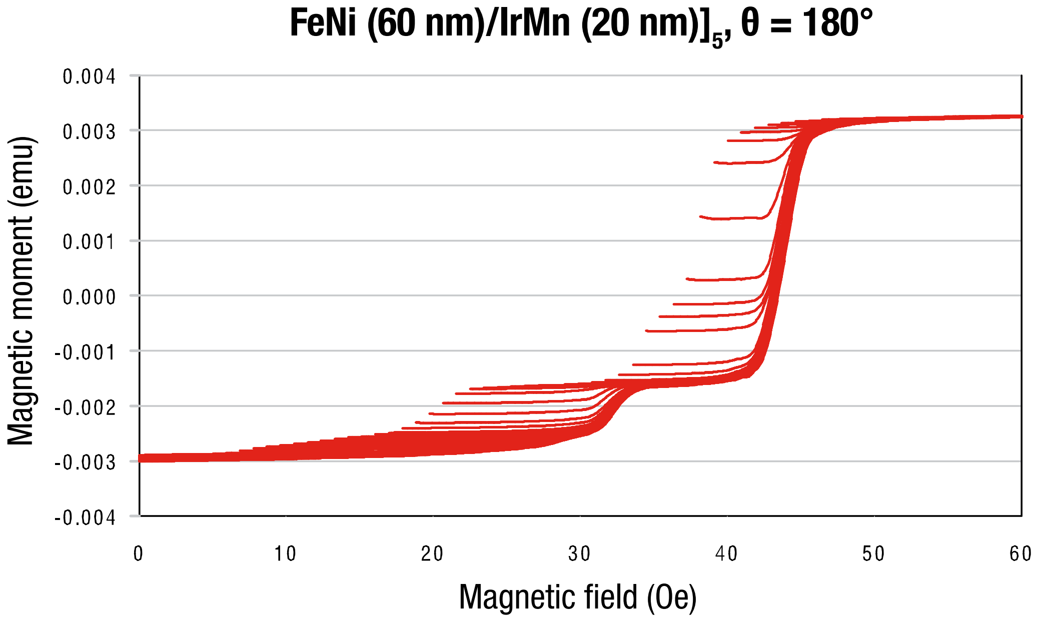

Figure 7. FORCs for [FeNi (60 nm)/IrMn (20 nm)]$_5$ at ϴ = 180°

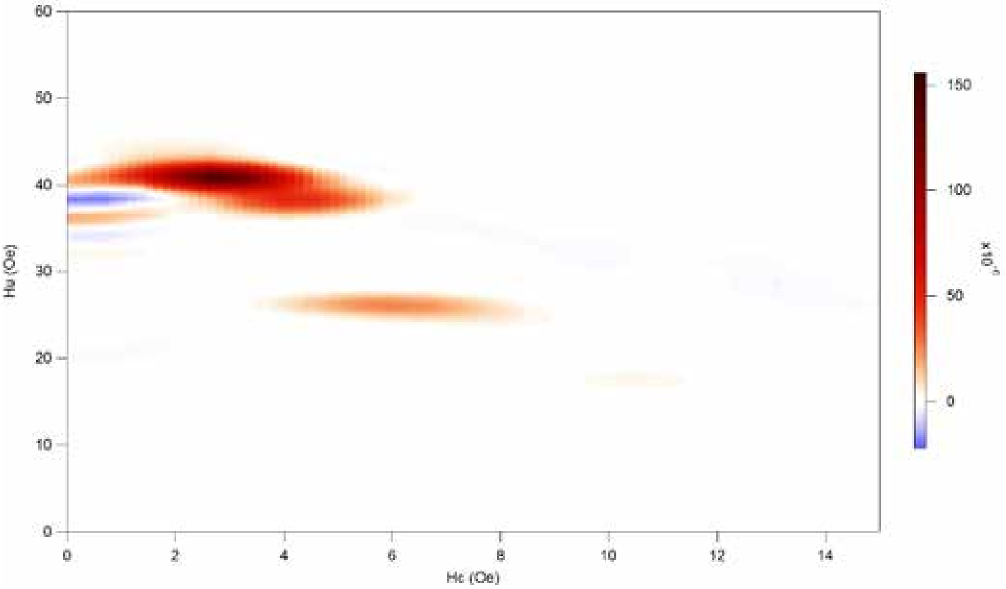

Figure 8. FORC diagram for [FeNi (60 nm)/IrMn (20 nm)]$_5$ at $\Theta = 180°$

Conclusion

While hysteresis loop measurements are sufficient for exploring the magnetic properties of many materials, they cannot be used to characterize interactions and coercivity distributions. The FORC measurement technique is indispensable in uncovering these characteristics which reveal insight into the relative proportions of reversible and irreversible components of the magnetization in many technologically important magnetic materials.

References

[1] First-Order-Reversal-Curve Analysis of Permanent Magnet Materials, B. C. Dodrill, Lake Shore Application Note, 2014

[2] First-Order-Reversal-Curve Analysis of Nanoscale Magnetic Materials, B. C. Dodrill, L. Spinu, Technical Proceedings of the 2014 NSTI Nanotechnology Conference and Exposition, CRC Press, Vol. 1, pg. 513, 2014

[3] First-Order-Reversal-Curve Analysis of Exchange Bias Magnetic Multilayer Films, B. C. Dodrill, L. Spinu, Lake Shore Application Note, 2014

[4] Lake Shore Cryotronics, USA

[5] Microsense, USA

[6] Quantum Design, USA

[7] Cryogenic Limited, UK

[8] R. J. Harrison and J. M. Feinberg, “FORCinel: An improved algorithm for calculating first-order reversal curve distributions using locally weighted regression smoothing”, Geochemistry, Geophysics, Geosystems, 9, 11, 2008

[9] Sample courtesy of L. Spinu, Advanced Materials Research Institute, University of New Orleans

[10] Sample courtesy of C. Garcia, Massachusetts Institute of Technology