Magnetometry Measurements of Nanomagnetic Materials

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ABSTRACT

Magnetometers are used to characterize magnetic material properties. The measurement most commonly performed to characterize a material's magnetic properties is that of a major hysteresis loop. More complex magnetization curves covering states with field and magnetization values located inside the major hysteresis loop, such as first-order-reversal-curves (FORC), can provide additional information that can be used to characterize magnetic interactions and coercivity distributions in magnetic materials. In this presentation, we will present a fast, high sensitivity (15 nemu RMS noise floor) electromagnet-based VSM that has been designed for characterizing nanomagnetic materials over a broad range of temperatures (4.2 K to 1273 K) and magnetic fields (>3.2 T), and we will present typical measurement results for various nanoscale magnetic materials.

Keywords: magnetometry, nanomagnet, hysteresis, forc, vibrating sample magnetometer

1 MAGNETIC MEASUREMENT TECHNIQUES

Magnetometry techniques can be broadly classified into two categories: inductive and force-based. The two most commonly used inductive techniques are vibrating sample (VSM) and superconducting quantum interference device (SQUID) magnetometry. Alternating gradient magnetometry (AGM) is the most often used force-based technique. Nanoscale magnetic materials (nanowires, nanoparticles, thin films, etc.) typically possess weak magnetic signatures, owing to the small amount of magnetic material that is present. Thus, one of the most important considerations in determining which type of magnetometer is best suited to specific materials is its sensitivity, as this determines the smallest magnetic moment that may be measured with acceptable signal-to-noise. Measurement speed, i.e., the time required to measure a hysteresis loop, is also important because it determines sample throughput, and it is particularly important for FORC measurements because a typical series of FORCs can contain thousands to tens of thousands of data points. The final consideration is the temperature and field range over which measurements are to be performed.

1.1 Vibrating Sample Magnetometry (VSM)

In vibrating sample magnetometry, originally developed by Simon Foner [1] 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 <2 K to 1273 K using integrated cryostats or furnaces.

Commercial VSM systems provide measurements to field strengths of ~3.4 T using conventional electromagnets [2, 3], as well as systems employing superconducting magnets to produce fields to 16 T [4, 5]. In an electromagnet based VSM a typical hysteresis loop measurement can take as little as a few seconds to a few minutes, and a typical series of FORCs takes minutes to hours. When used with superconducting magnets higher field strengths are possible which is necessary to saturate some magnetic materials, such as rare earth permanent magnets, however the measurement speed is inherently slower due to the speed at which the magnetic field can be varied using superconducting magnets. A typical hysteresis loop measurement can take tens of minutes or more, and a typical series of FORCs can take a day or longer. Magnetometers employing superconducting magnets are more costly to operate since they require liquid helium. Cryogen-free systems employing closed cycle refrigerators or liquefiers that recover helium in liquid helium based systems are available, but these represent an expensive capital equipment investment. The noise floor of commercially available VSMs is in the 10^{-7} to 10^{-8} emu range.

1.2 Superconducting Quantum Interference Device (SQUID) Magnetometry

Quantum mechanical effects in conjunction with superconducting detection 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, their noise floors are limited to 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 SQUIDs [4, 5]; therefore, the measurement is inherently slow due to the speed at which

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the magnetic field can be varied, as is the case for superconducting magnet-based VSM systems. A typical hysteresis loop measurement can take tens of minutes or more and a typical series of FORCs can take a day or longer.

1.3 Alternating Gradient Magnetometry (AGM)

Force methods involve determination of the apparent change in weight for a material when placed in an inhomogeneous magnetic field. 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 [2]. The AGM has a noise floor 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 and a typical series of FORCs takes minutes to hours. Commercial AGM systems can be used for ambient temperature measurements to the moderate ~3 T fields achievable with electromagnets.

2 ELECTROMAGNET-BASED VSM: SENSITIVITY AND SPEED

Figure 1 shows a schematic representation of an electromagnet-based VSM. A variable magnetic field is produced by an electromagnet. The VSM sensing coils are mounted on the pole faces of the magnet, and are balanced so as to produce zero signal (voltage) in the absence of a sample. A Hall probe, which is connected to a gaussmeter, is also mounted on the electromagnet pole face for closed-loop control of the magnetic field. A sample of any form (solid, powder, thin film, etc.) is affixed to the end of the VSM sample rod, which is in turn attached to the VSM head. The sample is vibrated in the z-direction within the sensing coils, and the resulting induced voltage in the sensing coils is passed through a preamplifier and then to a narrow bandwidth lock-in amplifier (LIA), which is tuned to the drive frequency of the VSM head.

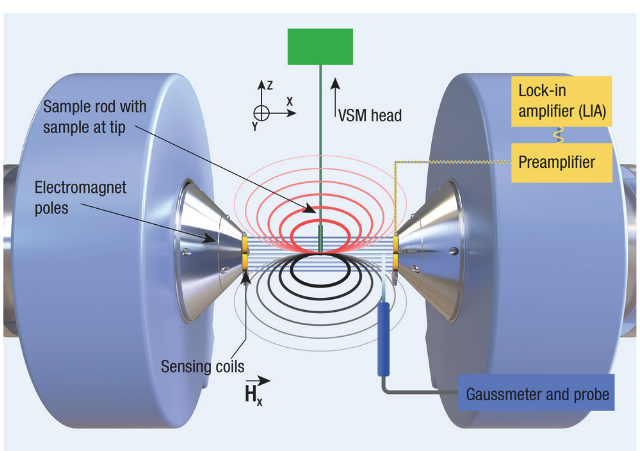


Figure 1: Schematic representation of a VSM.

A VSM's sensitivity depends on a number of factors:

- Electronic sensitivity.
- Noise rejection through signal conditioning.
- Amplitude and frequency of mechanical drive.
- Thermal noise of sensing coils.
- Optimized design and coupling (proximity) of sensing coils to the sample under test.
- Vibration isolation of the mechanical head assembly from the electromagnet and VSM sensing coils, and minimization of environmental mechanical and electrical noise sources, which can deleteriously effect VSM sensitivity.

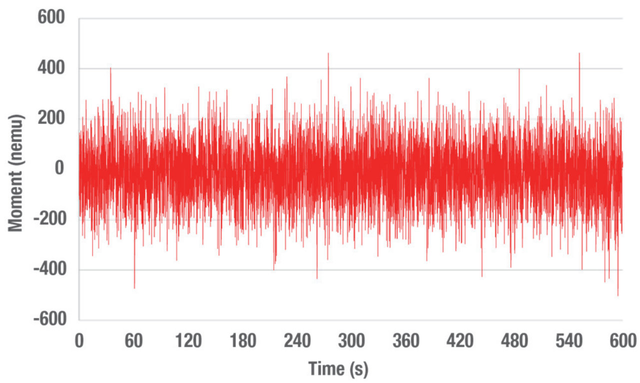
The voltage induced in the VSM sensing coils is given by:

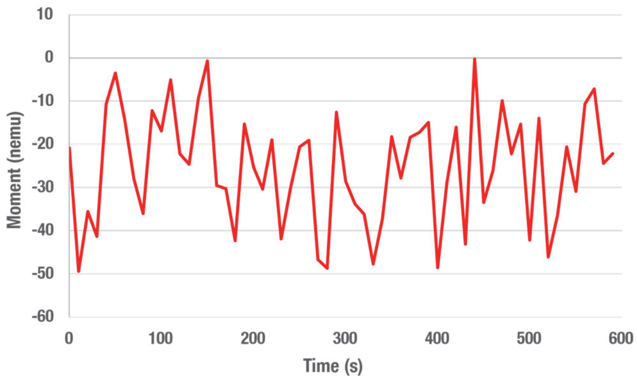
$$V_{emf} = mAfS \tag{1}$$

Where:
m = magnetic moment
A = amplitude of vibration
f = frequency of vibration
S = sensitivity function of VSM sensing coils.

It is clear from this equation that increasing A, f, or S will improve moment sensitivity. S may be increased by either increasing the coupling between the sense coils and the sample under test (i.e., minimize gap spacing), or by optimizing the design of the sensing coils (i.e., number of windings, coil geometry, etc.) And, of course, signal averaging also improves sensitivity. The data shown below were recorded at ambient temperature using a Lake Shore Model 8600 VSM [2]. In this system the head drive frequency is 83 Hz, the drive amplitude is variable between 0.064 mm and 6.4 mm peak-to-peak, and the control/measurement electronics and sensing coils have been optimized for characterizing nanomagnetic materials that possess weak magnetic signatures.

Figures 2 and 3 show typical noise measurement results at 100 ms/point (top) and 10 s/point (bottom) averaging. Note that the vertical axis is expressed in nemu (10^{-9} emu). The RMS noise values are noted in the figure caption.





Figures 2 and 3: Noise at 100 ms/point (figure 2, top) and 10 s/point (figure 3, bottom) averaging. The observed noise is 119.5 nemu and 13 nemu RMS, respectively.

The 8600 VSM has also been designed for fast measurements, providing field ramp rates to 1 T/s, and data acquisition as fast as 10 ms/point. Figure 4 shows typical low moment measurement results for a CoPt bit-patterned (bit size <100 nm) magnetic media (thin film) sample with saturation moment $m_{sat} = 20$ µemu (10^{-6} emu). The hysteresis loop was recorded for ±5 kOe in 25 Oe steps at 100 ms/point averaging. The total loop measurement time was 1 min 25 s. Figure 5 shows results for a synthetic antiferromagnetic thin film [Ta(2.5 nm)/Ru(5 nm)/Co(5 nm)/Ru(0.8 nm)/Co(5 nm)/Cu(6 nm)/Co(5 nm)/Ru(1.4 nm)/Co(10 nm)/Ta(5 nm)] with m_{sat} <2 µemu [6]. The hysteresis loop was recorded for ±500 Oe in 2.5 Oe steps at 2 s/point averaging. The total loop measurement time was 28 min.

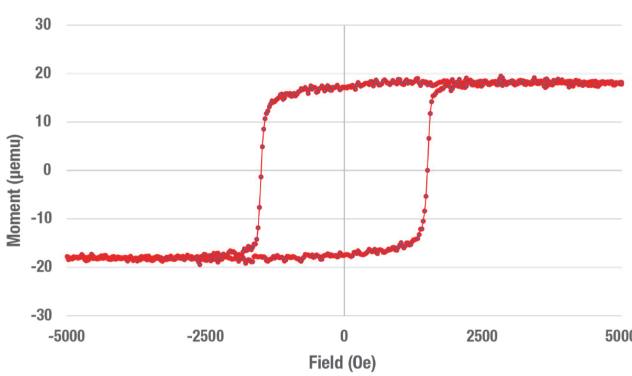


Figure 4: 1 min 25 s at 100 ms/point hysteresis loop for a 20 µemu CoPt nanomagnet array.

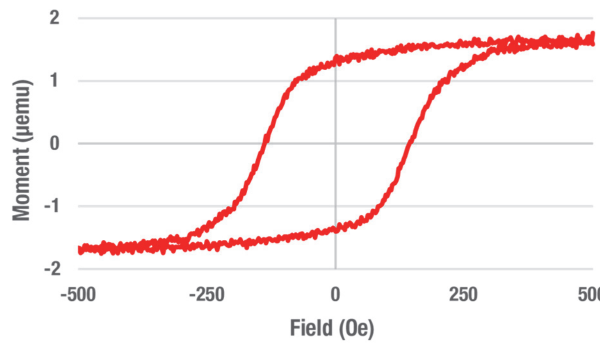


Figure 5: 28 min at 2 s/point hysteresis loop for a <2 µemu synthetic antiferromagnetic thin film.

3 FIRST-ORDER-REVERSAL-CURVE (FORC)

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 FORC distribution $\rho(H_a,H_b)$ is the mixed second derivative, that is, $\rho(H_a,H_b) = -(1/2)\partial^2 M(H_a,H_b)/\partial H_a \partial H_b$. A FORC diagram is a 2D or 3D 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 corresponds to the distribution of interaction or reversal fields, and H_c the distribution of switching or coercive fields.

FORC has been extensively used by earth and planetary scientists studying the magnetic properties of natural samples because FORC can distinguish between single-domain (SD), multi-domain (MD), and pseudo single-domain (PSD) behavior, and because it can distinguish between different magnetic mineral species [7, 8]. It has also been used to characterize interactions and coercivity distributions in magnetic recording media [9, 10], nanowire arrays [11], exchange-coupled permanent magnets [12], and exchange-biased magnetic multilayers [13]. Finally, while it is very difficult to unravel the complex magnetic signatures of multiphase magnetic materials from a hysteresis loop measurement alone, FORC can differentiate between phases in such materials [14, 15].

A typical series of FORCs can contain thousands to tens of thousands of data points, making the measurement very time consuming if the measurement speed of the magnetometer is slow. Figure 6 shows 100 FORCs (8818 data points) recorded at 500 ms/point averaging in 1 h and 20 min for the CoPt bit-patterned magnetic media (thin film) sample shown in figure 4. This is a fraction of the time that would be required if using a superconducting magnet-based VSM or SQUID system. Figure 7 shows the resultant 2D FORC diagram [16] where H_u corresponds to the distribution of interaction or reversal fields, and H_c the distribution of switching or coercive fields. The "boomerang" shape of the FORC distribution and the shift in the maximum towards negative interaction fields are features that are normally associated with exchange interactions [17] and thus these results suggest that exchange interactions are occurring between adjacent bits.

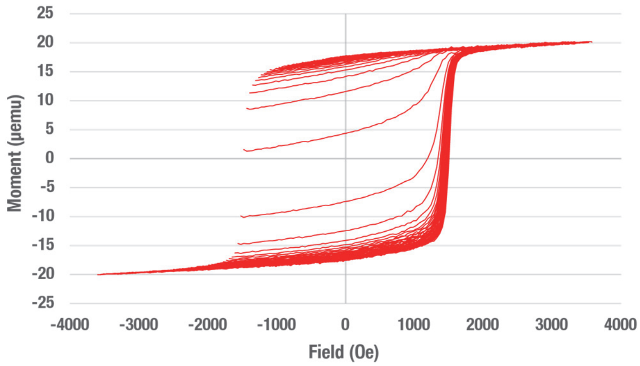


Figure 6: 1 h and 20 min measurement of 100 FORCs for a 20 µemu CoPt bit-patterned magnetic media (thin film).

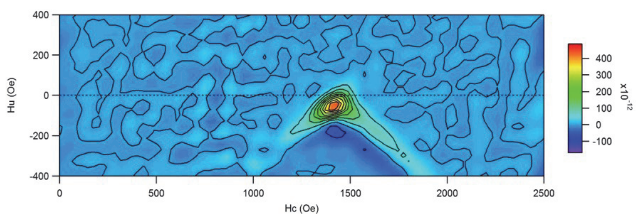


Figure 7: 2D FORC diagram for the CoPt bit-patterned magnetic media (thin film) sample.

4 SUMMARY

In this presentation we have discussed the advantages and disadvantages of the most commonly used inductive and force-based magnetometry methods, and presented an electromagnet-based VSM [2] optimized for characterizing nanomagnetic materials possessing weak magnetic signatures. We have presented measured RMS noise data as a function of signal averaging, and also typical hysteresis loop measurements for samples possessing saturation moments of <2 and 20 µemu. Results of fast hysteresis and low moment (20 µemu) FORC measurements have been presented as well, demonstrating the measurement speed of the VSM, which is particularly important in acquiring FORC data.

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