APPLICATION NOTE



Model 8610 High-Field (HF) VSM

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Introduction

Vibrating sample magnetometers (VSMs) provide the most commonly used technique to characterize magnetic materials. VSMs can measure the magnetic properties of magnetically soft (low coercivity) and hard (high coercivity) materials in many forms: solids, powders, single crystals, thin films, or liquids. They can be used to perform measurements from low to high magnetic fields employing electromagnets (EM) or high-field (HF) superconducting magnets (SCM). They can also be used to perform measurements from very low to very high temperatures with integrated cryostats or furnaces. Furthermore, they possess a dynamic range extending from 10⁻⁸ emu (10⁻¹¹ Am²) to above 10³ emu (1 Am²), enabling them to measure materials that are both weakly magnetic (e.g., ultrathin films, nanoscale structures) and strongly magnetic (permanent magnets).¹



The magnetic field range over which measurements are to be performed is dictated by the materials that are being measured. Permanent magnets are important materials for motors and generators that convert electricity to power and vice versa. Their importance has increased because of the recent drastic shift of vehicle powertrain from fossil fuel to electricity. Therefore, the demand for high-performance permanent magnets has increased significantly. Permanent magnet materials such as the rare earths (NdFeB, SmCo) and hard ferrites typically require large magnetic fields to saturate and demagnetize the materials. EM-based VSMs may be adequate, although HF SCM-based VSMs that can achieve fields to 90 kOe (9 T) or higher are sometimes required. If a permanent magnet material requires higher fields than are achievable in an EM-VSM, the sample is usually pre-magnetized using a pulsed magnetizer, and then the second quadrant demagnetization curve is measured in the EM-VSM. From the second guadrant demagnetization curve, the intrinsic magnetic properties of the material may be determined, i.e., the saturation magnetization M_s (or $4\pi M_s = B - H$), remanence M_r (or residual induction B_r), coercivity H_c , intrinsic coercivity H_{ci} , permeability (*B/H*) and maximum energy product BH_{max} .

This application note discusses the Model 8610 HF VSM and presents HF results for rare-earth sintered NdFeB and barium hexaferrite permanent magnet samples.



8610 HF VSM

The Model 8610 HF VSM incorporates a variable gap GMW² 10-inch EM configured with FeCo pole tips that provides maximum applied fields to >36 kOe (3.6 T). The 8610 features the same high sensitivity of 15 nemu (15×10^{-11} Am²) RMS noise at 10 s/pt averaging and high speed field ramp rate of 10 kOe/s (1 T/s) as Lake Shore's 8604 (4 in EM) and 8607 (7 in EM) VSM systems.

HF FeCo pole tips are optional for the 8604 and 8607 but are standard for the 8610. The maximum applied fields for the 8604, 8607, and 8610 HF VSMs at pole gaps 1, 2, 3, and 5 are shown in table 1.



Gap	Sample space	8604 high field	8607 high field	8610 high field
1 – Room temperature	3.5 mm	27.6 k0e (2.76 T)	32.2 k0e (3.22 T)	36.2 k0e (3.62 T)
2 – Room temperature	8 mm	25.2 k0e (2.52 T)	29.8 k0e (2.98 T)	33.7 k0e (3.37 T)
3 – Single-stage variable temperature (78 to 950 K)	16 mm*	20.3 k0e (2.03 T)	26.0 k0e (2.60 T)	29.8 k0e (2.98 T)
5 – Cryostat (4.2 to 450 K)/oven (308 to 1273 K)	24 mm*	15.5 kOe (1.55 T)	22.7 kOe (2.27 T)	26.3 kOe (2.63 T)

*Sample size 6.4 mm with VT options

Table 1: Maximum applied fields for the 8604, 8607, and 8610 HF VSMs as a function of pole gap



Figure 1: Initial magnetization curve and MHL for a NdFeB permanent magnet sample

8610 HF results

Sintered (powder) NdFeB

Figure 1 shows the initial magnetization curve and major hysteresis loop (MHL), and figure 2 shows the isothermal (IRM) and DC demagnetization (DCD) remanence curves for a sintered (powder) NdFeB magnet sample. The measurements were performed to applied fields of ± 36 kOe (3.6 T). Figure 3 shows the second quadrant demagnetization curve (from a previously magnetized state) for a NdFeB magnet sample.



Figure 2: IRM and DCD remanence curves for a NdFeB permanent magnet sample

While the most common measurement used to characterize a material's magnetic properties is the MHL, as illustrated in figure 1, more complex magnetization curves covering states with field and magnetization values located inside the major hysteresis loop, such as minor hysteresis loops and first-order-reversal-curves (FORCs), can give additional information that can be used for characterization of magnetic interactions in fine-particle magnetic materials^{3, 4} and also permanent magnet materials.^{5, 6, 7}

A FORC is measured by saturating a sample in a field H_{sat} , decreasing the field to a reversal field H_a , then measuring moment versus field H_b as the field is swept back to H_{sat} . This process is repeated for many values of H_a , yielding a series of FORCs as shown in figure 4 for the NdFeB permanent magnet sample with H_{sat} = 36 kOe (3.6 T). The FORC distribution $\rho(H_a, H_b)$ is the mixed second derivative:

 $\rho(H_a, H_b) = -(1/2)\partial 2 \ M(H_a, H_b)/\partial H_a \partial H_b$

A FORC diagram is a 2D or 3D contour plot of $\rho(H_a, H_b)$. It is common to change the coordinates from (H_a, H_b) to:

$H_c = (H_b - H_a)/2, H_u = (H_b + H_a)/2$

H_u represents the distribution of interaction or reversal fields, and *H_c* represents the distribution of switching or coercive fields of the hysterons. FORC analysis software packages such as FORCinel⁸ and VARIFORC⁹ are typically used to generate FORC diagrams from the measured FORCs, although the 2D FORC diagram for the NdFeB permanent magnet sample shown in figure 5 was generated using Lake Shore's RTForc[™] software.¹⁰ RTForc[™] calculates the FORC distributions and displays the FORC diagram in real-time, significantly reducing the time required to collect and analyze FORC data.



Figure 3: Second quadrant demagnetization curve for a NdFeB permanent magnet sample



Figure 4: Measured FORCs for a NdFeB permanent magnet sample



Figure 5: 2D FORC diagram for a NdFeB permanent magnet sample

Exchange-coupled barium hexaferrite (BaFe₁₂O₁₉) nanoparticles (NPs)

Figure 6 shows the initial magnetization curve and MHL, and figure 7 shows the IRM/DCD remanence curves for a sample consisting of nanometer-sized (~60 nm) barium hexaferrite BaFe₁₂O₁₉ exchange-coupled nanocomposite. The measurements were performed to applied fields of ± 36 kOe (3.6 T). There is a subtle "kink" in the MHL at low fields, suggesting the presence of a low and high coercivity phase. Figure 8 shows the measured FORCs with H_{sat} = 36 kOe (3.6 T), and figure 9 shows the resultant 2D FORC diagram.¹⁰ The FORC diagram shows two peaks corresponding to the low and high coercivity components, and the region between the two peaks is related to the exchange coupling between the two phases.¹¹



Figure 6: Initial magnetization curve and MHL for a $BaFe_{12}O_{19}$ permanent magnet sample



Figure 7: IRM and DCD remanence curves for a $BaFe_{12}O_{19}$ permanent magnet sample



Figure 8: Measured FORCs for a BaFe₁₂O₁₉ permanent magnet sample



Figure 9: 2D FORC diagram for a BaFe₁₂O₁₉ permanent magnet sample

Summary

In this application note, we discussed the Model 8610 HF VSM and presented HF (3.6 T) hysteresis, IRM/DCD, second quadrant demagnetization curve, and FORC measurement results for rare-earth NdFeB and exchange-coupled barium hexaferrite permanent magnet samples. HF SCM-based magnetometers may be required to fully saturate and therefore properly characterize some permanent magnet materials. However, such materials' second quadrant demagnetization curve may be characterized in an EM-based VSM provided the material is initially pulse magnetized. The magnetic field in EM-based VSMs can be swept at up to 10 kOe/s (1 T/s), and a typical hysteresis loop measurement can take as little as a few seconds to a few minutes; a typical series of FORCs takes minutes to hours. While SCM-based magnetometers provide higher field strengths, the measurement speed is inherently slower due to the speed at which the magnetic field can be varied using superconducting magnets due to their large inductance. Field sweep rates are typically limited to 200 Oe/s (20 mT/s); thus, a typical hysteresis loop measurement can take tens of minutes or more, and a typical series of FORCs can take a day or longer.



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