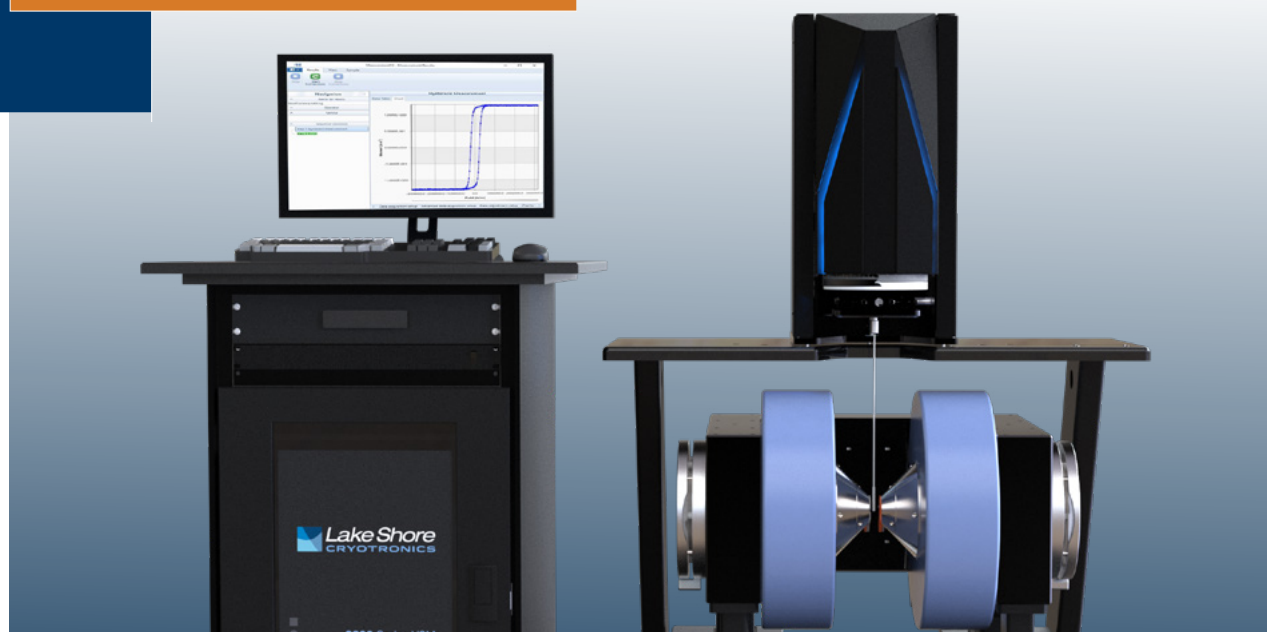




Characterizing Permanent Magnet Materials Using the 8600 Series VSM

Brad Dodrill, Cosmin Radu

Introduction

The vibrating sample magnetometer (VSM) is 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 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 10^3 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 magnet (PM) materials such as the rare-earths (NdFeB, SmCo) and hard ferrites typically require large magnetic fields to saturate, and to demagnetize the materials. EM-based VSMs may be adequate, although high-field SCM-based VSMs that are able to 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 quadrant 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} .

In this application note we will present 8600 VSM results for rare-earth sintered NdFeB and barium hexaferrite permanent magnet samples.

8600 VSM

The maximum applied fields for the 8604 and 8607 VSMs when configured with the high-field (HF) FeCo pole tips at pole gaps of 7.5 (1), 12 (2), 20 (3), and 28 (5) mm are tabulated in Table 1 (an optional configuration providing field strengths to >3.6 T is also available).

Gap	Pole gap	Sample access	8604 high field	8607 high field	VSM noise**
1 – Room temperature	7.5 mm	3.5 mm*	27.6 kOe (2.76 T)	32.2 kOe (3.22 T)	15 nemu
2 – Room temperature	12 mm	8 mm*	25.2 kOe (2.52 T)	29.8 kOe (2.98 T)	30 nemu
3 – Single-stage variable temperature	20 mm	16 mm*	20.3 kOe (2.03 T)	26.2 kOe (2.62 T)	90 nemu
5 – Cryostat/oven	28 mm	24 mm*	15.5 kOe (1.55 T)	23.1 kOe (2.31 T)	300 nemu

*Sample size 6.4 mm with VT options.

**10 s averaging time.

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



8600 results for permanent magnets

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 demag curve (from a previously magnetized state) for a NdFeB magnet sample.

While the most common measurement used to characterize a material's magnetic properties is measurement of 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^{2, 3}, and also permanent magnet materials.^{4, 5, 6}

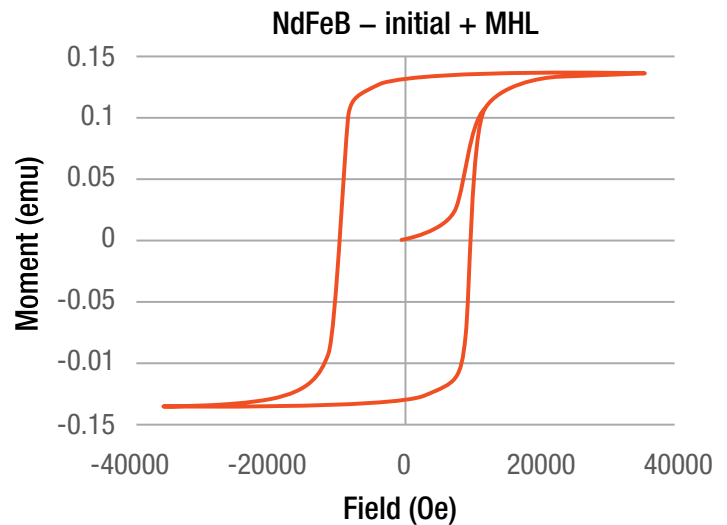


Figure 1: Initial magnetization curve and MHL for a NdFeB permanent magnet sample.

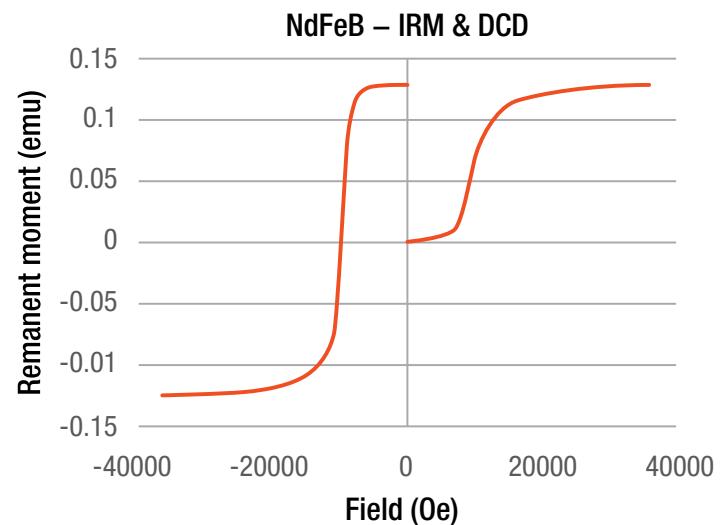


Figure 2: IRM and DCD remanence curves for a NdFeB permanent magnet sample.

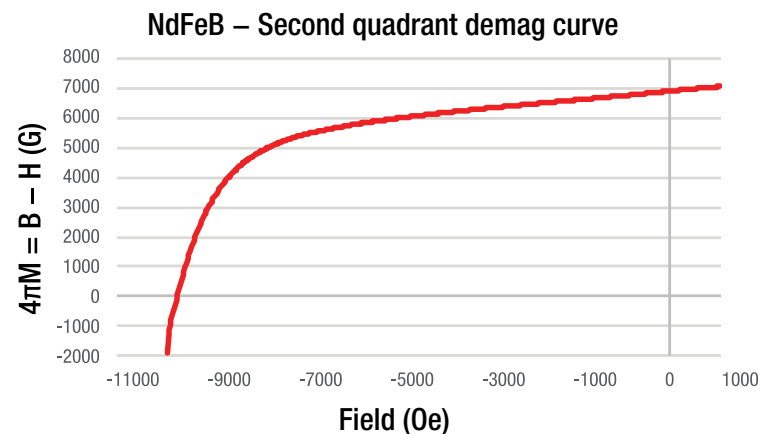


Figure 3: Second quadrant demag curve for a NdFeB permanent magnet sample.



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. The FORC distribution $\rho(H_a, H_b)$ is the mixed 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 FORCinel⁶ 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.⁹ RTForc[™] calculates the FORC distributions and displays the FORC diagram in real-time, which significantly reduces the time required to collect and analyze FORC data.

Exchange-coupled barium hexaferrite ($\text{BaFe}_{12}\text{O}_{19}$) nanoparticles (NPs)

There is currently considerable research underway in the development of rare-earth free or "lite" permanent magnet materials owing to supply chain vulnerabilities and environmental concerns.

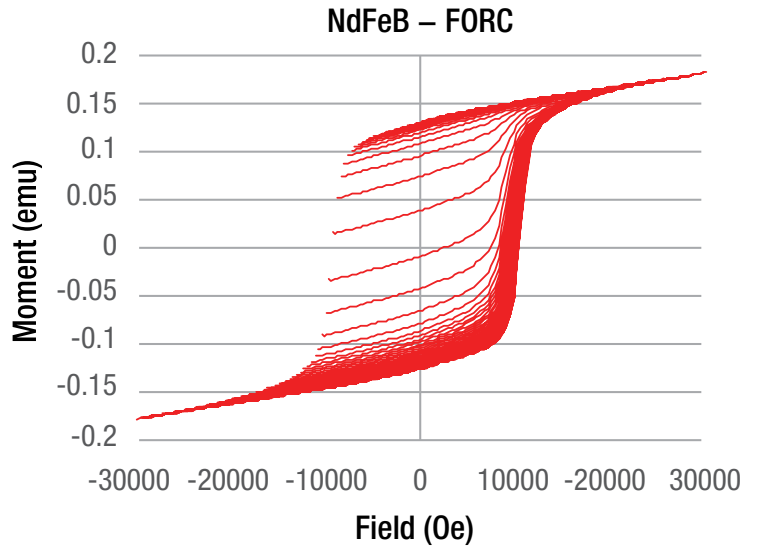


Figure 4: Measured FORCs for a NdFeB permanent magnet sample.

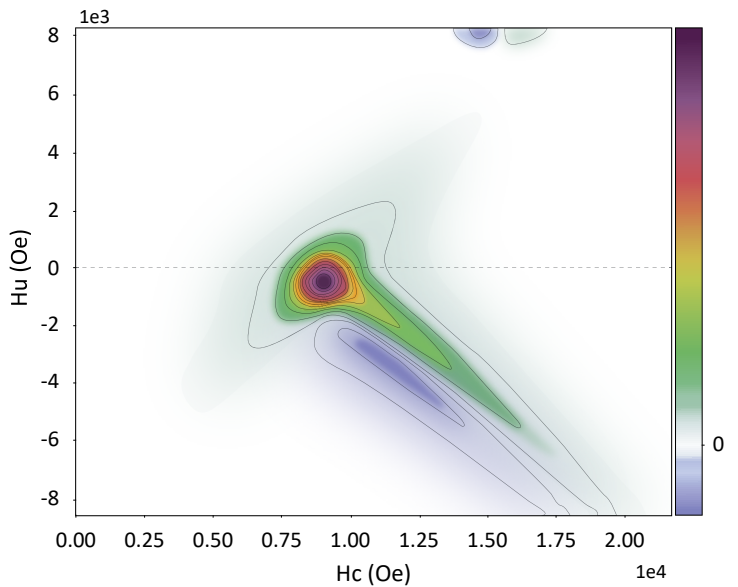


Figure 5: 2D FORC diagram for a NdFeB permanent magnet sample.



One approach being pursued is to exchange-couple a soft (low coercivity) high saturation magnetization ferromagnetic phase with a hard (high coercivity) low saturation magnetization ferromagnetic phase. This leads to a single-phase magnet with higher coercivity, remanence, and energy product than can be obtained from each individual phase magnet. Exchange coupled soft/hard or so called “exchange spring” permanent magnets possess magnetic properties comparable to rare-earth magnets with a nearly 80% reduction of rare-earth constituent materials, which reduces the cost of the magnet and mitigates supply chain vulnerabilities.

Figures 6 and 7 show the initial magnetization curve and MHL, and IRM/DCD remanence curves, respectively, for a sample consisting of nanometer-sized (~60 nm) barium hexaferrite $\text{BaFe}_{12}\text{O}_{19}$ 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 and 9 show the measured FORCs with $H_{\text{sat}} = 36$ kOe (3.6 T) and resultant 2D FORC diagram⁹, respectively. 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.¹⁰

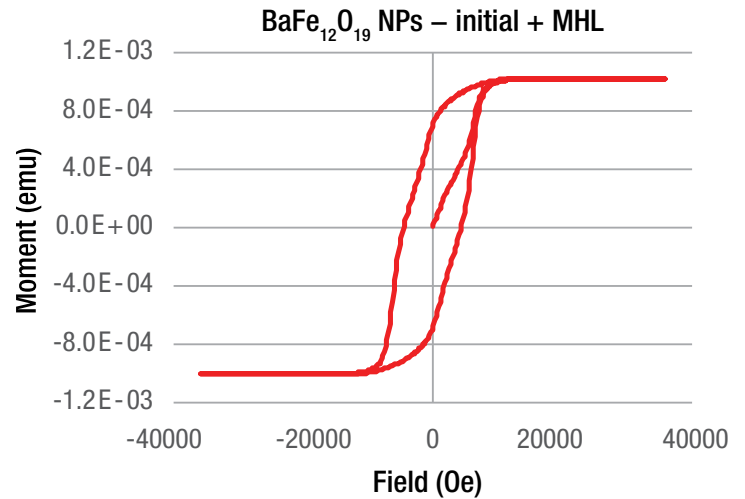


Figure 6: Initial magnetization curve and MHL for a $\text{BaFe}_{12}\text{O}_{19}$ permanent magnet sample.

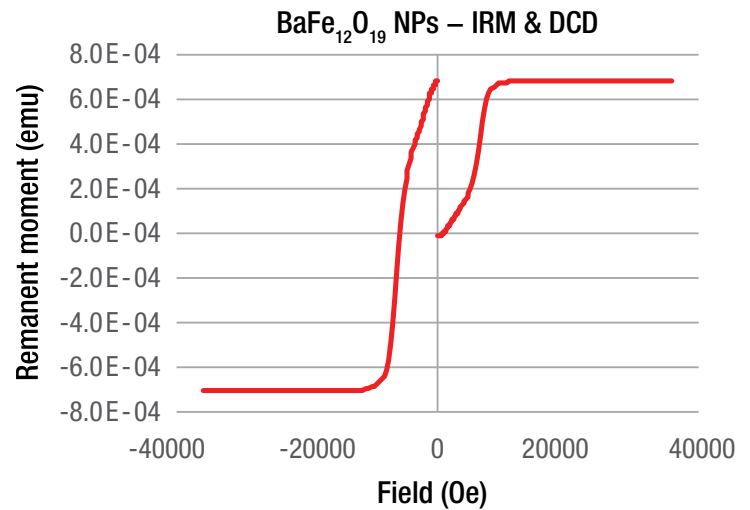


Figure 7: IRM and DCD remanence curves for a $\text{BaFe}_{12}\text{O}_{19}$ permanent magnet sample.

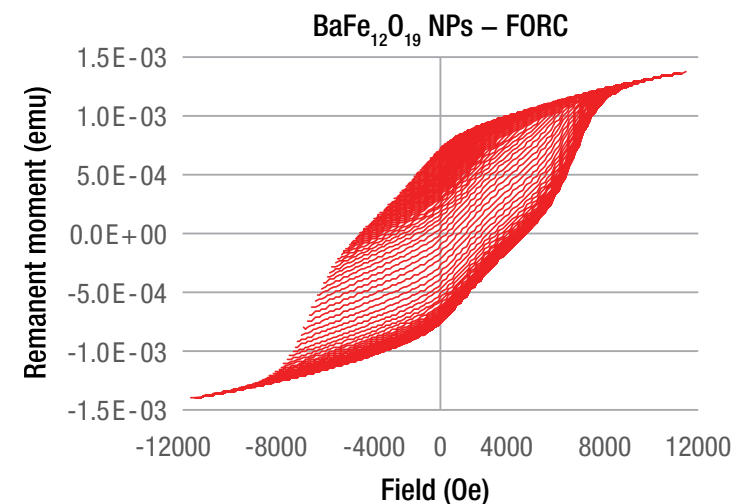


Figure 8: Measured FORCs for a $\text{BaFe}_{12}\text{O}_{19}$ permanent magnet sample.

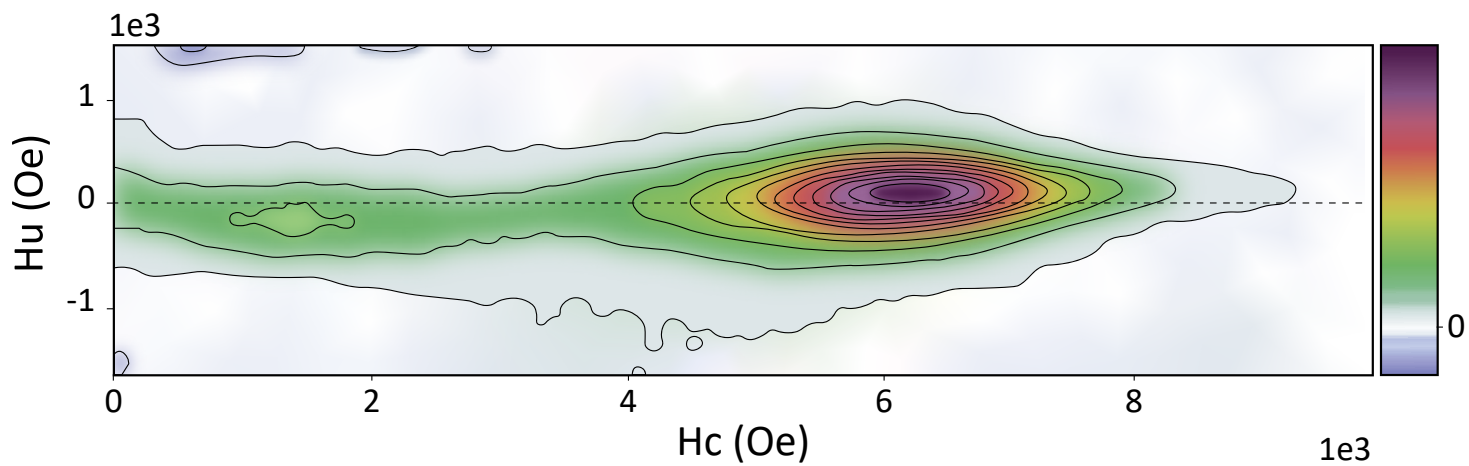


Figure 9: 2D FORC diagram for a $\text{BaFe}_{12}\text{O}_{19}$ permanent magnet sample.

Summary

In this application note we have discussed the use of the Model 8600 VSM in the characterization of permanent magnet materials and presented results for rare-earth NdFeB and exchange-coupled barium hexaferrite samples.



References

- ¹ B. C. Dodrill, J. R. Lindemuth, *Vibrating Sample Magnetometry*, in *Magnetic Measurement Techniques for Materials Characterization*, Springer Nature, edited by V. Franco, B. C. Dodrill, 2021.
- ² C. R. Pike, A. P. Roberts, K. L. Verosub, *Characterizing Interactions in Fine Magnetic Particle Systems Using First Order Reversal Curves*, *J. Appl. Phys.*, 85, 6660, 1999.
- ³ A. P. Roberts, C. R. Pike, K. L. Verosub, *First-Order Reversal Curve Diagrams: A New Tool for Characterizing the Magnetic Properties of Natural Samples*, *J. Geophys. Res.*, 105, 461, 2000.
- ⁴ S. Okamoto, *Permanent Magnets Materials*, in *Magnetic Measurement Techniques for Materials Characterization*, Springer Nature, edited by V. Franco, B. C. Dodrill, 2021.
- ⁵ T. Schrefl, T. Shoji, M. Winklhofer, H. Oezel, M. Yano, G. Zimanyi, *First Order Reversal Curve Studies of Permanent Magnets*, *J. Appl. Phys.*, 111, 07A728, 2012.
- ⁶ M. Pan, P. Shang, H. Ge, N. Yu, Q. Wu, *First Order Reversal Curve Analysis of Exchange Coupled SmCo/NdFeB Nanocomposite Alloys*, *J. Mag. Mag. Mat.*, 361, 219, 2014.
- ⁷ R. J. Harrison, 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.
- ⁸ R. Egli, *VARIFORC: An Optimized Protocol for Calculating Non-regular First-Order Reversal Curve (FORC) Diagrams*, *Global and Planetary Change*, 203, 110, 203, 2013.
- ⁹ B. C. Dodrill, H. S. Reichard, T. Shimizu, *Real-Time FORC (RTForc™) Software for the 8600 Series VSM*, Lake Shore Application Note, 2020.
- ¹⁰ Y. Cao, M. Ahmadzadeh, K. Xe, B. Dodrill, J. McCloy, *Multiphase Magnetic Systems: Measurement and Simulation*, *J. Appl. Phys.*, 123(2), 023902, 2018.

Copyright © Lake Shore Cryotronics, Inc. All rights reserved. Specifications are subject to change.

Lake Shore Cryotronics, the Lake Shore logo, the square graph logomark, and Cernox are registered trademarks of Lake Shore Cryotronics, Inc.

All other trade names referenced are the service marks, trademarks, or registered trademarks of their respective companies.

110425

Questions? Answers?

Visit <https://forums.lakeshore.com/>
and become part of the conversation!

