Measurements with a VSM

# Permanent Magnet Materials

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## Introduction

Permanent magnet materials are of growing interest to the electronics industry and to manufacturers of electro-mechanical devices in that they constitute indispensable elements in many electronic apparatus, circuits, electric motors, consumer, and defense products. The magnets have major influence on the size, efficiency, stability, and cost of these devices and systems.

The development of rare-earth magnet materials in the 1970's profoundly and positively influenced the application of permanent magnet materials owing to their large energy product and increased volume efficiency. The rare-earth alloy magnets that have been most extensively studied and developed are SmCo and NdFeB.

The presence of rare-earth metals in magnet alloys has a surprising influence on magnetic properties, and hence measurement of magnetic properties is exceedingly important in connection with research and development of new magnet materials. In a similar vein, magnetic measurements are utilized in magnet production and manufacturing environments to ensure quality in the end product and to provide critical process control feedback concerning the magnet manufacturing process itself.

This application note will discuss the utility of the Vibrating Sample Magnetometer (VSM) measurement methodology in the characterization of permanent magnet properties, with a particular emphasis on SmCo rare-earth magnet materials.

## Magnetism and the Permanent Magnet Process

A permanent or *hard* magnetic material retains magnetism or remanence even in the absence of an applied magnetic field and exhibits a large intrinsic coercivity, i.e., the field required to demagnetize the material. Alternatively, magnetically *soft* materials require application of an external field to exhibit useful magnetic properties, and typically exhibit very small coercivities. The demagnetization curve or 2$^{nd}$ quadrant of the M(H) or B(H) loop is the most often measured property that dictates a permanent magnets' suitability as a magnetic device. Its shape contains information concerning how the magnet will operate under static and dynamic conditions, and hence is a critical parameter in determining the suitability of the material for a specific application. The desirable qualities of a permanent magnet material are; high coercivity ($H_{ci}$), high saturation magnetization ($M_{sat}$ or $B_{sat}$), high remanence ($M_{rem}$ or $B_{rem}$), high energy product $(BH)_{max}$, and nearly linear 2$^{nd}$ quadrant B(H) characteristics.

Many permanent magnet materials are manufactured so that magnetic properties are enhanced along a preferred axis. This is realized if the crystal lattice structure of the material itself has preferred directions for alignment of the magnetic moments, and is referred to as magnetocrystalline anisotropy. Other permanent magnet materials are manufactured using processes that establish a net alignment of needle-shaped particles or platelets. These magnets base their properties on the shape anisotropy of the particles, where the shape of the particles produces an internal field which may differ from the applied field, leading to enhanced coercivity along the particle major axis.

The most widely used type of permanent magnet material is the class of ceramic ferrites which are made from iron oxide, $Fe_2O_3$. These are fine particle magnets produced by powder metallurgical methods. They exhibit high coercivities and nearly linear demagnetization curves, and are particularly well suited for many applications, including electric motors. The magnetism of ferrites is founded in the magnetocrystalline anisotropy of the particles.

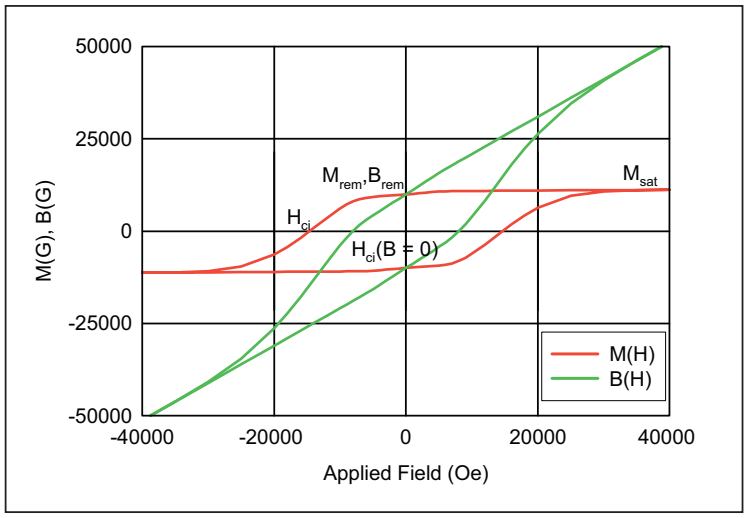


***Figure 1*** *M(H) and B(H) curves for a sintered NdFeB sample. These data were recorded using a superconducting magnet-based VSM.*

Alnico magnets represent another commonly used permanent magnet material. In these materials, elongated magnetic particles are precipitated throughout the matrix of an Al-Ni-Fe-Co alloy during the manufacturing process. The magnetism of these materials is due to the shape anisotropy of the particles.

It became apparent in the 1960's that attempts to further enhance or improve the magnetic properties of ferrites and alnico magnets were exhausted. The search then began for other materials with high uniaxial magnetocrystalline anisotropy, high coercivity, and high saturation magnetization. Rare-earth magnet alloys represented the most promising candidates, and advances in the development of these magnetic materials over the last 30 years have had a profound, and far-reaching impact on magnetic devices.

The rare-earth magnet materials that have gained the most attention include alloys of $SmCo_5$, $Sm_2Co_{17}$, and $Nd_2Fe_{14}B$. It was believed that the cost and availability of the principle constituents in SmCo would limit their commercial success, so in the 1980's considerable effort was expended to replace scarce Co with abundant Fe in combination with rare-earth metals, ultimately leading to the development of NdFeB magnets.

The question of whether Sm or Nd alloys are preferred depends critically on the specific application to which the magnet material will be used. Where the energy density of Nd alloys is superior to the Sm alloys, the Sm alloys' strength is in its' thermal stability. For applications where the temperature is stable and to just above room temperature, NdFeB is a better choice. Conversely, for those applications where the temperature may vary significantly, or the magnet material may be subjected to elevated temperature environments, SmCo is a better choice.

## The Hysteresis Loop: M(H) and B(H)

A typical full or major hysteresis loop for a NdFeB sintered magnet is illustrated in figure 1, where both the magnetization M and magnetic induction B are presented. Some of the pertinent parameters extracted from such loop measurements are indicated in the figure. In the context of permanent magnet materials, the 2$^{nd}$ quadrant is the most pertinent in connection with the utilization of a magnet material in a magnetic device

The Vibrating Sample Magnetometer (VSM) is based on vibrating a sample in a magnetic field to produce an alternating emf within a set of suitably placed pick-up or sensing coils. This induced emf is directly proportional to the magnetic moment m or magnetization M of the sample under test. VSM's are used widely since magnetic properties can be measured for a diverse range of sample sizes and configurations, i.e., powders, solids, single crystals, thin films, and liquids, and because they are particularly well suited to allow measurements at both low and high temperatures. In the development of rare-earth magnet materials the VSM has been used extensively to measure saturation, remanence, coercivity, anisotropy fields, etc., and also to measure temperature dependent parameters of interest, for example Curie temperatures. The VSM is an "open loop" measurement in that the sample and field source (e.g., electromagnet) do not constitute a closed flux line loop. Hence the measured parameters must be corrected for demagnetization effects to yield the true intrinsic material parameters most often of interest for permanent magnet materials. In the open loop measurement, the internal field experienced by a sample differs from the applied field owing to an internal demagnetizing field which depends on the sample geometry under test.

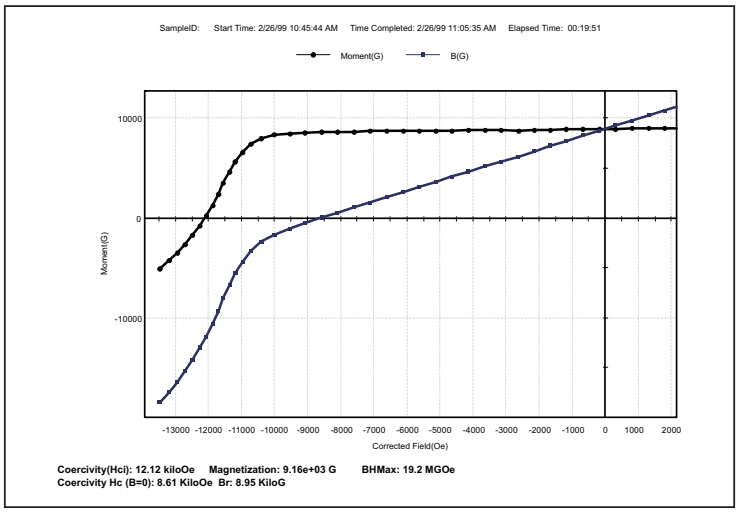


***Figure 2*** *M(G) and B(G) vs. $H_{int}$ for a $Sm_2Co_{17}$ magnet material at 25 °C.*

The VSM measures magnetic moment m as a function of applied field $H_{applied}$. The equations that relate open loop (VSM) to closed loop (BH looper) measurements are (cgs-units):

Eq. 1) $M(G) = 4pM = 4pm/V$

where M is the volume magnetization in units of emu/cc, and V = sample volume. The internal magnetic field is related to the applied field via,

Eq. 2) $H_{int} = H_{applied} - 4pN_DM$

where $N_D$ = sample demagnetization factor (in SI units), and M is as defined in eq. 1. Values for $N_D$ may be determined empirically or analytically, and there are countless references containing demagnetization corrections for various sample geometries. The magnetic induction B and magnetization M are related via,

Eq. 3) $B = H_{int} + 4pM = H_{applied} + 4pM(1 - N_D).$

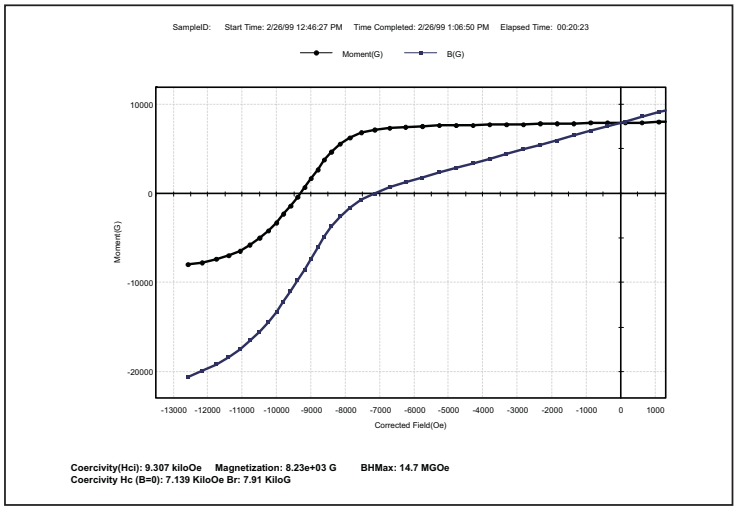


***Figure 3*** *M(G) and B(G) vs. $H_{int}$ for a $Sm_2Co_{17}$ magnet material at 300 °C.*

Provided $N_D$ values can be determined with reasonable accuracy, the VSM measurement can be used to derive the B(H) curve which is measured directly in the closed loop measurement.

Closed loop measurements (BH loopers and hysteresisgraphs being the most common) allow freedom from demagnetization corrections and directly measure the magnetic induction B. The usual practice is to place a magnetic sample between the pole pieces of an electromagnet. The change in flux density of the sample is determined by integration of the induced voltage in a search coil that is wrapped around the sample using a fluxmeter. The sample is in physical contact with the electromagnet pole caps, thus constituting a closed circuit, and therefore eliminating the need for demagnetization corrections since the internal and applied fields are identical.

Regardless of which technique is used, since the portion of the hysteresis loop that is most often of interest is the demagnetization curve or 2nd quadrant, it is common to first magnetize a sample specimen to saturation using a magnetizer, and then measure only the 2nd quadrant M(H) or B(H) characteristics. The magnetization M measured in a VSM may be converted to induction B. Thus, the VSM yields information essentially identical to that acquired in a closed loop measurement if demagnetization corrections are made. The advantages the VSM provides over closed loop measurement techniques are: magnetization M is measured directly, full loop characteristics can be measured to very high fields using superconducting magnet technology, a wider array of sample configurations can be accommodated, and variable temperature measurements are more readily adapted to the VSM configuration. This latter capability is of particular merit in connection with magnetic property measurements of permanent magnet materials since a knowledge of thermal stability versus demagnetization is requisite to application specific magnet design. The remainder of this application note will present VSM M(H) and B(H) data for SmCo samples, and will compare these results with closed-loop (hysteresisgraph) data.

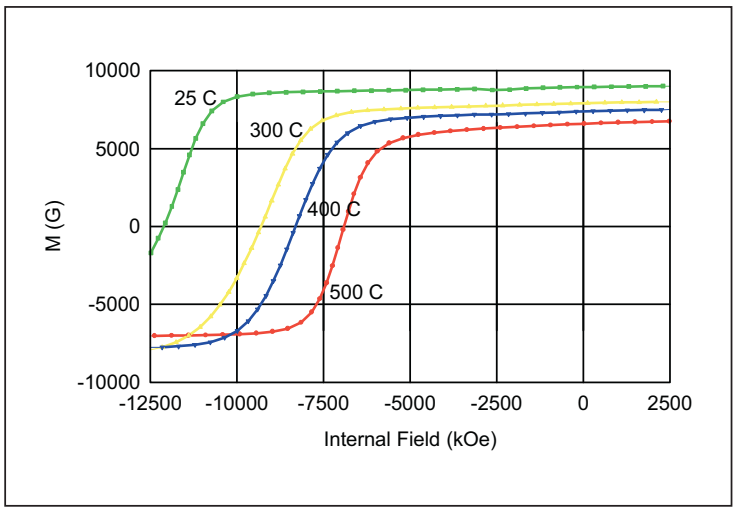


***Figure 4*** *M(H,T) curves for the SmCo magnet material.*

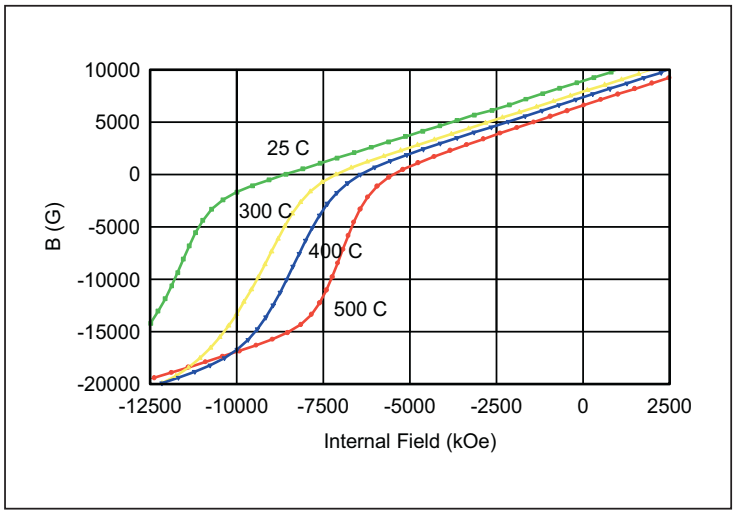


***Figure 5*** *B(H,T) curves for SmCo magnet material.*

## $Sm_2Co_{17}$: Measurements With a VSM

To compare results obtained using the open loop VSM technique with closed-loop methodologies, demagnetization curves were measured for $Sm_2Co_{17}$ samples at ambient and elevated temperatures, and compared with hysteresisgraph data.

To effect demagnetization corrections, magnet samples were fabricated with a right circular cylinder geometry, from which demagnetization factors $N_D$ could be determined with reasonable accuracy. The samples were oriented such that the cylinder axis was at right angles to the applied magnetic field. For this particular sample geometry and field orientation, $N_D$ »» 0.3 (in SI units). The sample was initially magnetized to saturation, and then the 2nd quadrant M(H) characteristics measured in the VSM at room temperature (i.e., 25 °C). The $M(H_{applied})$ data was then converted to $B(H_{int})$ using equations 2 and 3 above to determine; $H_{ci}$, $H_c(B = 0)$, $B_{rem}$, and $BH_{max}$.

A VSM furnace assembly, which provides variable temperature capability to 1000 °C, was used to conduct M(H,T) measurements at 300, 400, and 500 °C. At these elevated temperatures, applied fields of only 15 kOe were sufficient for saturation, hence the sample's M(H) characteristics were measured from +15 kOe to -15 kOe for both saturation, and measurement of the resultant demagnetization curve.

Figures 2 and 3 illustrate results of the VSM measurements at 25 °C and 300 °C, respectively. Both M(G) (eq. 1), and B(G) (eq. 3) are plotted together as a function of the internal (demagnetization corrected) field (eq. 2).

Tabulated below are the magnet performance parameters vs. temperature. Figures 4 and 5 show the family of M(H,T) and B(H,T) curves respectively, and graphically illustrate the thermal degradation of magnet properties with increasing temperature.

| T(°C) | $B_r$(kG) | $H_{ci}$(kOe) | $BH_{max}$(MG Oe) | $H_c(B = 0)$ (kOe) |
|---|---|---|---|---|
| 25 | 8.95 | 12.12 | 19.2 | 8.61 |
| 300 | 7.91 | 9.307 | 14.7 | 7.139 |
| 400 | 7.38 | 8.311 | 12.7 | 6.438 |
| 500 | 6.59 | 6.911 | 9.78 | 5.466 |

To compare performance between the VSM and closed-loop measurement techniques, another $Sm_2Co_{17}$ sample (cubic geometry) was measured employing both a VSM and a hysteresisgraph. The sample measured exhibits an energy product in excess of 30 MG Oe, and an intrinsic coercivity near 30 kOe at room temperature. Since neither the VSM or hysteresisgraph used in this comparative investigation were capable of producing fields of this magnitude, measurements were conducted at 300 °C, where $H_{ci}$ values were within both instruments' field range capabilities. The VSM data are shown in Figure 6, and the results of both measurements are tabulated below. These results agree to the few percent level, and thus demonstrate that the VSM is capable of producing results essentially identical to the closed-loop hysteresisgraph measurement.

| | VSM | Hysteresisgraph | % Variation |
|---|---|---|---|
| $B_r$(kG) | 10.6 | 10.38 | + 2.1% |
| $H_{ci}$(kOe) | 7.573 | 7.78 | -2.7% |
| $H_c(B = 0)$ | 6.795 | 6.80 | < 0.1 % |
| $BH_{max}$(MG Oe) | 24.1 | 23.42 | 2.9% |

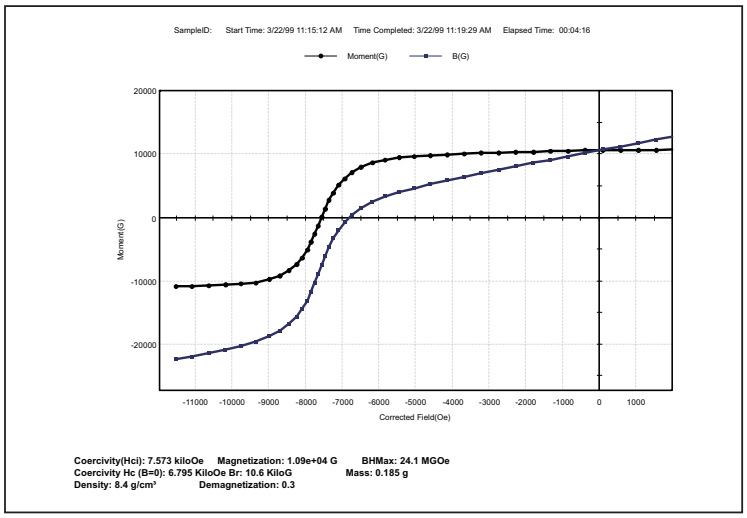


***Figure 6*** *M(G) and B(G) at 300 °C for a high energy product (>30 MG Oe) SmCo magnet material.*

## Intrinsic Coercivity: Rate Dependence

In comparing measured coercivity values between various, or even identical measurement methodologies, it is important to ensure that the field ramp rate is identical. Many magnetic materials (particularly dispersions) exhibit an intrinsic coercivity which is dependent on field ramp rate, i.e., $H_{ci}$ µln(dH/dt). This property has been extensively studied for certain types of magnetic materials, e.g., particulate recording media, thin film media materials (hard disks), etc.

In producing SmCo magnet materials, the constituent powders are mixed together, a reduction/diffusion process is applied to segregate the desired SmCo stoichiometry, and then the particles are milled to the desired final size, typically 5 - 10 mmm. The materials are multi-phase with complicated microstructures, and therefore also exhibit rate dependent characteristics.

Tabulated below are $H_{ci}$ values vs. field ramp rate, dH/dt, for a SmCo alloy. These results are illustrated graphically in Figure 7 where $H_{ci}$ vs. ln(dH/dt) is presented. It's clear that if measurement comparisons between open-loop/closed-loop, or between identical measurement techniques are attempted the field ramp rate must be similarly identical.

| dH/dt (Oe/s) | $H_{ci}$ (kOe) |
|---|---|
| 20 | 6.76 |
| 25 | 6.777 |
| 33.3 | 6.796 |
| 50 | 6.829 |
| 75 | 6.862 |
| 200 | 6.965 |

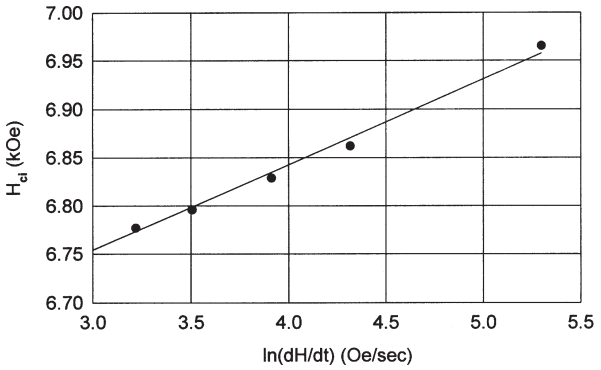


***Figure 7*** *$H_{ci}$ vs. ln(dH/dt) for a SmCo magnet sample.*

## Conclusions

This application note has discussed the utility of the Vibrating Sample Magnetometer (VSM) measurement methodology for characterizing the demagnetization curve properties of permanent magnet materials. Open-loop (VSM) and closed-loop (hysteresisgraph) results have been compared, and agreement to the few percent level has been demonstrated, provided sample demagnetization corrections are employed. The effect of field ramp rate on the intrinsic coercivity has been discussed, and the VSM's capability for investigating elevated temperature characteristics of permanent magnet materials has also been demonstrated.

## Acknowledgments

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## References

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