M-H Characteristics and Demagnetization Resistance of Samarium-Cobalt Permanent Magnets to 300 C

Janis M. Niedra
Sverdrup Technology, Inc.
Lewis Research Center Group
Brook Park, Ohio

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ABSTRACT

The influence of temperature on the M-H demagnetization characteristics of permanent magnets is important information for the full utilization of the capabilities of samarium-cobalt magnets at high temperatures in demagnetization-resistant permanent magnet devices. In high temperature space power converters, such as free-piston Stirling engine driven linear alternators, magnet demagnetization can occur as a long-term consequence of thermal agitation of domains and of metallurgical change, and also as an immediate consequence of too large an applied field. This paper investigates the short-term demagnetization resistance to applied fields derived from basic M-H data. This quasistatic demagnetization data was obtained for commercial, high-intrinsic-coercivity, Sm$_2$Co$_{17}$-type magnets, from 5 sources, in the temperature range 23 to 300 C. An electromagnet driven, electronic hysteresigraph was used to test the 1-cm cubic samples. The observed variation of the 2nd quadrant M-H characteristics was a typical rapid loss of M-coercivity and a relatively lesser loss of remanence with increasing temperature. The 2nd quadrant M-H curve knee point is used to define the limits of operation safe against irreversible demagnetization due to an excessive bucking field for a given flux density swing at temperature. Such safe operating area plots are shown to differentiate the high temperature capabilities of the samples from different sources. For most of the samples their 2nd quadrant M-H loop squareness increased with temperature, reaching a peak or a plateau above 250 C.

INTRODUCTION

Temperatures exceeding 250 C are now planned for the exciting magnets in high temperature power converters, such as the free-piston Stirling engine driven linear alternators [1], for multyear missions in space. There the need to minimize system mass and volume requires that the capabilities of the magnets be fully known and utilized up to some set margin of safety. Among the well known demagnetizing influences are mechanical shock, thermal agitation of domains over their pinning potential barriers [2] as well as thermally induced metallurgical change, an externally applied bucking field, and possibly radiation effects [3]. However, this paper is restricted to the study of the limits of operation safe against immediate and irreversible demagnetization due to combinations of high temperature and applied demagnetizing field. The basic data presented are the measured 2nd quadrant M-H characteristics at selected temperatures to 300 C of samarium-cobalt magnets from 5 manufacturers. Comparative plots of the decrease with temperature of the remanent magnetic moment ($M_r$) and of the M-coercivity ($\mu H_c$) are then shown. The knee-field ($\mu H_c$) is invoked to create plots that help to assess the margin of safety against irreversible loss of $M_r$ due to a given swing ($\Delta B$) below remanence of the magnetic induction at a given temperature.

Of the various presently available high-energy permanent magnet materials, only the samarium-cobalt type can produce magnets that retain at 300 C a useful remanence ($B_r \approx 0.9T$) and a sufficiently high intrinsic coercivity ($\mu H_c \geq B_r$) to avoid self-demagnetization and to keep the induction ($B$) roughly linear with the applied $H$ in the 2nd quadrant [4]. This investigation is further restricted to the "second generation" Sm$_2$Co$_{17}$-type magnets because these generally outperform the SmCo$_5$-type by the various magnet performance criteria such as $(BH)_{max}$ and $\mu H_c$.

APPARATUS AND PROCEDURES

The quasistatic, 2nd quadrant M-H characteristics of the precisely sized 1-cm cubic magnet samples were measured by an electromagnet-driven hysteresigraph. This instrument is an improved, 300 C model of a temperature control oven and probe coil assembly for magnet testing that was originally developed at the University of Dayton [5]. Briefly, the magnet sample fits closely between the flat and parallel faces of iron-cobalt pole pieces that are rigidly mounted to an electrically heated aluminum housing comprising the oven. These pole pieces couple the applied demagnetizing field ($H_d$) from the electromagnet to the sample. The B-flux is sensed by a coil surrounding the sample and the H-flux is sensed by an air coil adjacent to the B-coil. The signals from these coils are electronically integrated and combined so as to yield signals proportional to the intrinsic magnetic moment ($M$) within the B-coil as well as the $H_d$. A diagram of the apparatus and further details of construction are given in reference [6].
Absolute calibration of the M-axis at room temperature was referenced to a pure nickel standard in the form of a 1-cm cube having a known magnetization of 6.100 kG in an applied field of 10 kOe at 25 C. Up to about 200 C, the H-axis calibration was transferred from a precision reference magnet by a Hall effect probe.

Prior to measurement, each sample was pulse magnetized in a charger coil at 100 kOe peak field. For measurements at 200 C and above, a sample was then preheated for 5 or so minutes to about 100 C below the measurement temperature in order to minimize chipping caused by thermal shock. Next the sample was inserted into the preheated fixture, allowed to stabilize for a few minutes to final temperature, and then the demagnetization curve was taken. To limit any ageing effects, no sample was ever soaked at measurement temperature for more than 10 minutes. Since the probe coil fixture, which holds the sample and the thermocouple, suffers unavoidable loss of heat during its removal from the oven when resetting integrators, the accuracy of magnet temperature reading was likely no better than ±3 C.

HIGH TEMPERATURE DEMAGNETIZATION DATA

Following the above procedure, 2nd quadrant M-H curves were taken for 10-sample groups of high $\mu$H$_r$, anisotropic 2-17-type magnets from 5 manufacturers. A representative sample was selected from each group, as the variation of characteristics within each group was usually smaller than the variation between groups. Figures 1a-1e present the basic data taken at selected temperatures from room to 300 C and Figure 1f is included to review definitions and illustrate the relations between M and B and $\Delta$B, in cgs units, pertinent to the data.

At room temperature, the remanence B$_r$ varies from 10.4 to 10.9 kG, whereas the $\mu$H$_r$ varies from 17.4 kOe to values exceeding the 32 kOe maximum field capability of the apparatus. At 300 C, the corresponding ranges are 9.1 to 9.7 kG and 7.2 to 10.0 kOe. Thus, all samples are seen to exhibit the usual rapid loss of M-coercivity and a relatively lesser loss of remanence as temperature increases. The room temperature M-H curve shape varies considerably according to sample source, partly due to the considerable differences in the $\mu$H$_r$; however, there is a tendency to increased squareness with rising temperature, as will be discussed.

It is also clear from Figure 1 that the room temperature M-H data is not always a reliable indicator of performance at 300 C. However, the samples with the highest room temperature $\mu$H$_r$ also exhibit the highest temperature coefficient $d(\mu H_r)/dT$. This is apparent from the plots of $\mu H_r$ versus temperature given in Figure 2, which in particular shows a clear reversal of the relative magnitudes of $\mu H_r$ of the Recoma (Re) and Shin-Etsu (SE) samples at about 220 C. From 23 to about 300 C, the normalized rates $d(\mu H_r/\mu H_r(23 C))/dT$, in %/C, are 0.28, 0.27, 0.29 and 0.18 for the IGT, EE, Re and SE samples, respectively. Presumably something in the processing or composition distinguishes the SE sample by its low rate of coercivity loss with temperature. Although it is generally believed that high coercivity in 2-17-type magnets is critically dependent on domain wall pinning by the alloy microstructure, no attempt is made here to relate observations to the complicated issues in coercivity and temperature. Further comments on coercivity may be found in reviews by Wallace [4], Gaunt [7], Kumar [8], and references therein.

The rate of loss of the remanence B$_r$ (=4$\pi$M$_r$) with increasing temperature is seen to be roughly 4.3 G/C for the 4 samples plotted in Figure 3. Data irregularities seen there are of a magnitude of the measurement repeatability for the apparatus. No loss of either B$_r$ or $\mu$H$_r$ that is irrecoverable by remagnetization, which is indicative of a metallurgical change, could be resolved after these short-term tests to 300 C; that is, the curves shown in Figures 1a-e were repeatable.

SAFE OPERATING AREA

M-H Knee Point and Irreversible Demagnetization

The point on the M-H characteristic in its 2nd quadrant where M=0.9M$_r$ or "knee point", is conventionally taken as the point of onset of rapid downslope. Depending on the M-H curve squareness, an immediate and significant loss of magnetization can set in if the applied field drives M below its knee point. This loss is then recoverable only by remagnetization. Or in magnetics design terminology, as temperature increases, the knee field ($\mu$H$_r$) decreases in magnitude and will eventually appear as a knee in the 2nd quadrant of the B-H characteristic illustrated in Figure 1f. Thus for magnets operating on a low-lying, static load line (e.g. B/H~--0.5) or else on a dynamic load line with a fixed swing $\Delta$B of B below B$_r$, an increasing temperature will force the knee point to cross to the right of the load line, with immediate and possibly catastrophic loss of remanence. Even if operation is confined to the right of the knee point, close approach to the knee is likely, from energy considerations, to increase the rate of magnetic ageing due to thermal agitation of domain walls over their pins. Early experiments confirming this effect in samarium-cohald magnets have been reported by Bachmann [9] and by Martin and Benz [10]. From these considerations, the variation of the knee point with temperature is taken to be important information for the design of demagnetization-resistant permanent magnet devices.

Using the IGT and Re magnets as examples, Figure 4 presents plots, derived from the data in Figure 1, that help a designer to assess the margin of safety against demagnetization due to too large an applied bucking field.
The plot of $|\mu H_k|$, showing its decrease with temperature, forms the boundary to the safe operating area (SOA). Superimposed on this plot are curves showing, at a given temperature, the magnitude $|H_d|$ of the demagnetizing field needed to produce a desired induction swing $\Delta B$ below $B_r$; these curves are parametrized by $\Delta B/B_r$, where $B_r$ is at temperature. The peak $H_d$ is found from the constitutive relation $B=H+4\pi M(H)$, at temperature, by setting $B=B_r-\Delta B$, as illustrated in Figure 1f. According to preceding remarks, $|H_d|<|\mu H_k|$ is necessary for safe operation to the right of the knee. For example, Figure 4a shows that the IGT sample can be safely operated up to at most 280 C with $\Delta B=0.9B_r$. The SOA plot in Figure 4b shows that the Re magnet is limited to 60 C for $\Delta B=0.9B_r$. From the point of view of such $\Delta B$-T SOA defined by the knee-field criterion, the IGT and Re samples were found to be the most and least resistant to demagnetization for temperatures from room to 300 C.

M-H Squareness

Squareness of the M-H characteristic in its 2nd quadrant is at least a qualitative measure of the amount of loss of magnetization in case M is driven below $M_k$ by $|H_d|>|\mu H_k|$. For a measure of squareness we shall consider the simple quotient $S=\mu H_k/\mu H$, in preference to the more complicated "fullness factor" $F=(\mu H)_{max}/(\mu H)$. Note that a perfectly square loop has $S=F=1$. A loop with vertical sides and a sloping top and bottom, which is a parallelogram and hence not strictly square, can have $S=1$; however, in such a case the consequences of $|H_d|>|\mu H_k|$ are the same as for a square loop.

Figure 5 compares S for the IGT, EE, Re and SE samples at the selected measurement temperatures. For all samples except the SE, the S increases with temperature above room, reaching a weak peak or plateau above 250 C. For the exceptional SE magnet, the S decreases gradually with temperature to 300 C. Plots of F not shown here have a similar behavior, but with less resolution among the samples. No correlation was found between ordering by squareness and ordering by SOA.

SUMMARY OF RESULTS AND CONCLUSIONS

Quasistatic demagnetization curves were obtained from room temperature to 300 C for 10-sample groups of high coercivity, anisotropic Sm$_2$Co$_17$-type magnets from 5 manufacturers. This data was taken using an electromagnet-driven, permanent magnet hysteresigraph based on induction sensing of the magnetization and field of 1-cm cubic samples held snugly between the faces of tapered iron-cobalt pole pieces mounted within a temperature control oven. Fields up to 32 kOe could be applied to a sample.

A representative sample was chosen from each group and its 2nd quadrant M-H curves at selected temperatures to 300 C are presented in Figure 1. For these samples the remanence $\mu M_r$ varied from 10.4 to 10.9 kG at room temperature and from 9.1 to 9.7 kG at 300 C. Their intrinsic coercivity $\mu H_c$ varied from 17.4 to over 32 kOe and from 7.2 to 10.0 Koe at these temperature extremes. The fractional change in coercivity with temperature was, as expected, much greater than that of the remanence. For 3 of the samples, this coercivity temperature coefficient was about 0.28%/C, whereas one sample had an exceptionally low 0.18%/C. The loss of remanence was roughly 4.3 G/C for these samples. Since no sample was ever soaked at measurement temperature for more than 10 minutes, no significant alloy microstructure aging was expected and indeed, the data curves were repeatable.

The resistance of these samples to short-term demagnetization was evaluated by a comparison, at a given temperature, of the knee field $\mu H_d$ needed to reduce the magnetization M to M, by the demagnetizing field below its remanence $B_r$. A condition necessary for safe operation was assumed to be $|H_d|<|\mu H_k|$, which simply states that one must not drive M below the knee point. Based on this criterion, composite plots versus temperature of $\mu H_d$ and $\mu H_k$ for a specified $\Delta B$ are shown to be useful for providing comparative overviews of the temperature-induction swing limits of safe magnet operation. Such safe area plots are presented for the samples found most and least resistant to demagnetization. At say 280 C, the most resistant sample can tolerate a $\Delta B$ up to 0.9$B_r$, as compared to only 0.6$B_r$ for the least resistant one. Furthermore, these safe area plots show that room temperature performance, such as a high $\mu H_d$, is not a reliable predictor of performance at high temperatures because slopes with temperature and their variability (especially for $\mu H_d$) may be considerable.

Since the amount of magnetization loss resulting from an excursion into an unsafe region depends on the M-H loop squareness in its 2nd quadrant, a brief study of a simple measure of squareness, namely $S=\mu H_k/\mu H$, is presented. For all but the sample with the exceptionally low coercivity temperature coefficient, S is shown to increase with temperature above room, reaching a weak peak or plateau above 250 C. The S of the exceptional sample decreases gradually with temperature from room to 300 C. Further study of squareness is suggested because the use of 2-17-type magnets above 250 C may force magnet operation close to the knee point of maximal squareness.

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REFERENCES


Figure 1. Demagnetization characteristics of commercial anisotropic 2-17 type samarium-cobalt magnets at selected temperatures to 300 °C, with illustration of definitions.
Figure 2. Temperature dependence of intrinsic coercivity for 2-17-type magnets from 4 sources. The normalized rates are about 0.28 and 0.18 %/C.

Figure 3. Temperature dependence of the remanence for 2-17-type magnets from 4 sources. The loss rate is about 4.3 G/C for all.

Figure 4. Temperature variation of the knee field ($M_{H_k}$) and the demagnetizing field ($H_d$) for selected induction swings ($\Delta B$) of 2-17-type magnets from 2 sources. The safe operating area is below the $M_{H_k}$ curve.

Figure 5. Temperature dependence of the squareness ($S$) for 2-17-type magnets from 4 sources.
The influence of temperature on the M-H demagnetization characteristics of permanent magnets is important information for the full utilization of the capabilities of samarium-cobalt magnets at high temperatures in demagnetization-resistant permanent magnet devices. In high temperature space power converters, such as free-piston Stirling engine driven linear alternators, magnet demagnetization can occur as a long-term consequence of thermal agitation of domains and of metallurgical change, and also as an immediate consequence of too large an applied field. This paper investigates the short-term demagnetization resistance to applied fields derived from basic M-H data. This quasistatic demagnetization data was obtained for commercial, high-intrinsic-coercivity, Sm2Co17-type magnets, from 5 sources, in the temperature range 23 to 300 °C. An electromagnet driven, electronic hysteresigraph was used to test the 1-cm cubic samples. The observed variation of the 2nd quadrant M-H characteristics was a typical rapid loss of M-coercivity and a relatively lesser loss of remanence with increasing temperature. The 2nd quadrant M-H curve knee point is used to define the limits of operation safe against irreversible demagnetization due to an excessive bucking field for a given flux density swing at temperature. Such safe operating area plots are shown to differentiate the high temperature capabilities of the samples from different sources. For most of the samples their 2nd quadrant M-H loop squareness increased with temperature, reaching a peak or a plateau above 250 °C.