IN-33
37385
p-8

NASA Technical Memorandum 104508

Makeup and Uses of a Basic Magnet Laboratory for Characterizing High-Temperature Permanent Magnets

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Prepared for the
First International High Temperature Electronics Conference
cosponsored by Sandia National Laboratories, Wright Research
and Development Center, and the University of New Mexico
Albuquerque, New Mexico, June 16–20, 1991

NASA

(NASA-TM-104508) MAKEUP AND USES OF A BASIC MAGNET LABORATORY FOR CHARACTERIZING HIGH-TEMPERATURE PERMANENT MAGNETS (NASA) 8 p
N91-30427
CSCL 09A
Unclas
G3/33 0037385

MAKEUP AND USES OF A BASIC MAGNET LABORATORY FOR CHARACTERIZING HIGH-TEMPERATURE PERMANENT MAGNETS

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E-6373

SUMMARY

A set of instrumentation for making basic magnetic measurements was assembled in order to characterize high-intrinsic-coercivity, rare-earth permanent magnets with respect to short-term demagnetization resistance and long-term aging at temperatures up to 300 °C. The major specialized components of this set consist of a 13-T-peak-field, capacitor-discharge pulse magnetizer; a 10-in.-pole-size, variable-gap electromagnet; a temperature-controlled oven equipped with iron-cobalt pole piece extensions and a removable paddle that carries the magnetization and field sensing coils; associated electronic integrators; and standards for field intensity H and magnetic moment M calibration. A 1-cm cubic magnet sample, carried by the paddle, fits snugly between the pole piece extensions within the electrically heated aluminum oven, where fields up to 3.2 T can be applied by the electromagnet at temperatures up to 300 °C. A sample set of demagnetization data for the high-energy Sm_2Co_{17} type of magnet is given for temperatures up to 300 °C. These data are reduced to the temperature dependence of the M–H knee field and of the field for a given magnetic induction swing, and they are then interpreted to show the limits of safe magnet operation.

INTRODUCTION

Temperatures approaching 300 °C are now planned for the exciting magnets to be used in free-piston-Stirling-engine-driven linear alternators for multiyear missions in space (Slaby 1989), where a high heat rejection temperature permits a great reduction in radiator size. Of the various presently available high-energy permanent magnet materials, only the samarium-cobalt type of material can produce magnets that retain at 300 °C a useful remanence ($B_r \sim 0.9$ T) and a sufficiently high intrinsic coercivity (${}_MH_c > B_r$) to keep the magnetic induction B roughly linear with the applied field H in the second quadrant (Wallace et al. 1989, Potenziani II et al. 1985, NMAB 1985, and Strnat 1983).

However, determining the characteristics of the SmCo type of magnet is not easy. First, these modern rare-earth–cobalt magnets are not easily magnetized; the "charging" field pulse must exceed the magnet's ${}_MH_c$ (usually tens of kilo-oersteds at room temperature) by at least a factor of 2 or 3 in order to develop the maximum possible coercivity. Further, at 300 °C significant instrumentation problems related to thermal expansion of the magnet sample temperature-control fixture, field-sensing coil expansion and wire insulation, and sample handling must be overcome. Such experimental difficulties have no doubt contributed to the scarcity of high-temperature demagnetization data on otherwise promising high-temperature magnets.

In this report we describe a set of basic magnetic measurement instrumentation for characterizing the demagnetization resistance of high-coercivity rare-earth permanent magnets at temperatures to 300 °C . We provide examples of data obtained using this setup, give interpretations, and indicate directions of future research.

INSTRUMENTATION AND PROCEDURES

For charging 1-cm cubic magnet samples we are presently using a pulse magnetizer of the mercury-ignitron-switched, capacitor-discharge type. A maximum of 24 kJ can be stored at 2000 V on our 0.012-F capacitor bank. This bank can be connected to either one of two solenoids, one providing peak fields up to 13 T in a 1-in. bore and the other up to 10 T in a 1.75-in. bore. Spatial nonuniformity of the field is less than 10 percent over a 2-in.-long central volume of these solenoids. The damping of this resistance-inductance-capacitance system is just sufficient to ensure a nonreversing pulse of about 10-ms full width at half maximum that rises rapidly to its peak in 2 ms and then tails off slowly in about 40 ms. This field is sufficient to develop fully the coercivity of even the hardest neodymium-iron-boron (NdFeB) type of magnet and is "overkill" for samarium-cobalt magnets.

The core instrument in our magnet testing laboratory is the magnet sample temperature-control fixture. It has a removable aluminum paddle that carries the sample along with built-in magnetization and field-sensing coils. The present instrument is an improved, 300 °C, model of a temperature-control and probe coil assembly for magnet testing that was originally developed at the University of Dayton (Mildrum and Graves 1981). Figure 1 shows how a precision 1-cm cubic sample fits closely between the flat and parallel faces of iron-cobalt pole pieces that are precision mounted to the electrically heated aluminum housing and together make up the temperature-controlled oven. These high-magnetic-saturation pole pieces serve as extensions of the electromagnet pole pieces; they are thermally insulated from the latter by 30-mil-thick Teflon shims. The driving electromagnet has a 10-in. pole size and a variable gap and is equipped with tapered pole caps. The fixture has tolerances and controlled overall thermal expansion such as to keep the effects of the sample-to-poleface gap negligible. The *B*-flux is sensed by a coil surrounding the sample, and the *H*-flux is sensed by another coil, of nearly the same area-turns and thermal expansion, located adjacent to the *B*-coil. After electronic integration the signals from these coils are subtracted so as to air-flux compensate the *B*-coil, resulting in a signal proportional to the intrinsic magnetic moment *M* within the *B*-coil. As the field provided by the electromagnet is slowly varied, the electronically processed signals from the two sensing coils yield the second quadrant *M–H* demagnetization curve, with possibly some extension into the third quadrant. Salient features of this high-temperature model are the high-saturation, high-curie-point, 50 percent iron-cobalt pole pieces; the use of ceramic-insulated sensing coil wire wound on machinable ceramic forms; and a bronze liner that provides a bearing surface for the sliding paddle. Above 200 °C and without this liner the aluminum-on-aluminum sliding contact was found to result in rapid surface gouging and even seizure of the paddle in its slot after only a few uses.

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 of the demagnetization characteristic, the samarium-cobalt samples were pulse magnetized in the charger coil at 100-kOe peak field. For measurements at 200 °C and above a sample was then preheated for approximately 5 min to about 100 deg C below the measurement temperature in order to minimize chipping caused by thermal shock. Next the sample was inserted into the preheated fixture and allowed to stabilize for a few minutes to final temperature, and then the demagnetization curve was taken. In order to limit any aging effects, no sample was ever soaked at measurement temperature for more than 10 min. Because 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 a magnet temperature reading was probably no better than ±3 deg C. In this regard, quick handling of hot samples becomes an important consideration. The best solution has been to let the sample be held by its own field to the face of a threaded Teflon spacer attached to a steel screw. The sample is attracted to the embedded screw, and the protruding part of the screw serves as a handle.

SUMMARY OF EXPERIENCE WITH SAMARIUM-COBALT MAGNETS

The initial investigations have been concentrated on the modern Sm_2Co_{17} type of material, because its energy product $(BH)_{max}$, as well as possibly other magnet performance criteria, generally exceeds that of the older $SmCo_5$ type of magnet. Figure 2 is typical of the variation of demagnetization characteristics with temperature that was observed for a commercial, anisotropic 2-17 type of magnet. It exhibited the usual rapid loss of coercivity and relatively lesser loss of remanence as temperature increased. The 24 °C curve terminates at $H \approx -32$ kOe because that is the maximum field strength achievable in the apparatus.

Data extending into the third M–H quadrant (not shown) were taken but became unreliable as M approached saturation. In fact, the $|M|$-signal was actually seen to decrease for sufficiently high $|H|$! Poleface saturation is believed to be the cause of this anomaly. Using the electrostatic analogy, one can see that the magnetic charges appearing at the ends of the sample magnet are normally canceled by image charges appearing in the contiguous pole faces. However, as the pole faces approach saturation, imaging effectiveness decreases, and a sample in a state of high M becomes subject to an increasing self-demagnetizing field in addition to the externally applied H. Then the M and H readouts are no longer valid for the sample. As yet, the authors know of no effective way to circumvent this limitation in this type of instrument, which prevents one from obtaining a complete hysteresis loop of a high-coercivity magnet. In light of this, certain vibrating sample magnetometers that are subject to image fields (Hoon and Willcock 1988) may also be susceptible to errors from variable imaging.

We now discuss ways that raw demagnetization data, as in figure 2, can be reduced to plots that exhibit the margin of safety against demagnetization in given circumstances of magnet use. Briefly, if a magnet is subject to an excessive demagnetizing field that drives M below the knee of the M–H curve (i.e., below the 10-percent-down-from-remanence point), one may expect an immediate and significant loss of magnetization. The amount of this loss, which is recoverable only by remagnetization, is obviously sensitive to the squareness of the M–H curve in the neighborhood of its knee. Also, from considerations of domain wall energy, domain wall pinning potentials, and thermal agitation (Mildrum et al. 1974), close approach to the knee is likely to accelerate the rate of the long-term remanence loss called magnetic aging. 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. Figure 3 presents such a plot of the knee field $|{}_MH_k|$, showing its decrease with temperature. 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 ΔB below remanence B_r. According to the preceding remarks, one clearly needs $|H_d| < |{}_MH_k|$ for safe operation to the right of the knee. For example, figure 3 shows that this magnet with $\Delta B = 0.9B_r$ cannot be safely operated above 280 °C. Considering the temperature range of 25 to 300 °C, figure 3, as well as similar data for other samples not presented herein, also shows that room-temperature performance is not a reliable predictor of performance near 300 °C because slopes with temperature, as well as their variability, can be considerable.

CONCLUSIONS AND FUTURE DIRECTIONS

This work has shown that an electromagnet-driven permanent magnet hysteresigraph based on induction sensing of the magnetization and field of 1-cm cubic samples held snugly between tapered iron-cobalt pole pieces can be practicable up to 300 °C for obtaining the second quadrant *M–H* demagnetization data of high-energy magnets such as samarium cobalt. The two sensing coils, wound with ceramic-insulated wire on machinable ceramic bobbins, have provided reliable and reproducible output even after numerous excursions to 300 °C. At room temperature the 3.2-T maximum field in the 1-cm gap between the iron-cobalt pole pieces was adequate to drive the magnetic moment to zero for all except the magnetically hardest samples; above 100 °C this limitation disappeared. In the third *M–H* quadrant and at high magnetization the data provided by this instrument can be erroneous because pole face saturation above about 2 T leads to magnetic decoupling (failure of magnetic charge imaging) of the sample from the pole faces. A vibrating sample magnetometer using spherical samples may have a wider range in this respect as well as in temperature. However, the 1-cm cubic sample has proven to be ideal for availability, ease of handling, and ease of magnetic charging.

The present hysteresigraph is thought to be suitable also for short-term (~100 hr) one-at-a-time aging tests of magnet samples at temperature and at a specified static demagnetizing field. For longer term (~5000 hr) aging there may be little choice but to simultaneously test multiple samples in an inert-gas chamber. Subjecting samples to a continuous controlled bucking field, other than the self-demagnetization in free space, and in-place testing at uninterrupted temperature may not be feasible for long-term aging tests.

ACKNOWLEDGMENTS

This research was sponsored by the NASA Lewis Research Center under the High Capacity Power element of the Civilian Space Technology Initiative.

REFERENCES

Hoon, S.R.; and Willcock, S.N. (1988) The Direct Observation of Magnetic Images in Electromagnet Vibrating Sample Magnetometers. J. Phys. E. Sci. Instrum., vol. 21, no. 5, pp. 480–487.

Mildrum, H.F., et al. (1974) An Investigation of the Aging of Thermally Prestabilized Sintered Samarium-Cobalt Magnets. IEEE Trans. Magn., vol. MAG–10, no. 3, Mar., pp. 723–725.

Mildrum, H.F.; and Graves, Jr., G.A. (1981) High Speed Permanent Magnet Generator Material Investigations—Rare Earth Magnets. Air Force Wright Aeronautical Laboratories, AFWAL-TR-81-2096, Oct.

NMAB (1985) *Magnetic Materials*, National Materials Advisory Board, NMAB-426, National Academy Press, Washington, DC.

Potenziani, E., II, et al. (1985) The Temperature Dependence of the Magnetic Properties of Commercial Magnets of Greater than 25 MGOe Energy Product. J. Appl. Phys., vol. 57, no. 8, pt. IIB, Apr. 15, pp. 4152–4154.

Slaby, J.C (1989) Free-Piston Stirling Technology for Space Power. NASA TM-101956.

Strnat, K. (1983) Study and Review of Permanent Magnets for Electric Vehicle Propulsion Motors. DOE/NASA/0189-83/2, NASA CR-168178.

Wallace, W.E., et al. (1989) Synthesis of High Energy Magnet Materials: Coercivity Mechanism, Surface Studies and New Alloys. Mater. Sci. Eng. B, vol. 3, no. 4, Sept., pp. 351–354.

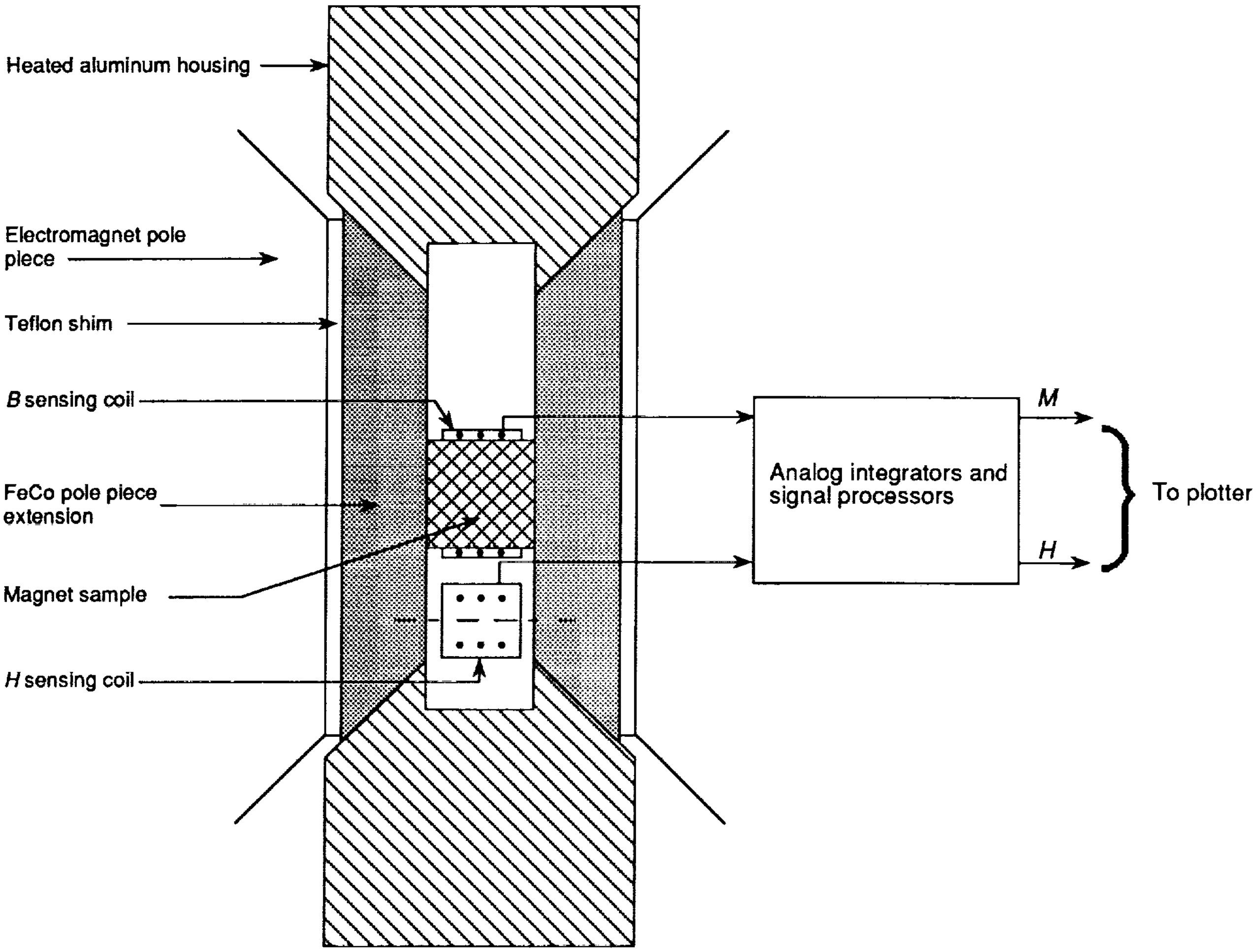

Figure 1.—Electromagnet-driven permanent magnet hysteresigraph. The FeCo pole pieces are an integral part of the temperature-control fixture. Electronic processing converts the (*B*, *H*) signals to (*M*, *H*) signals.

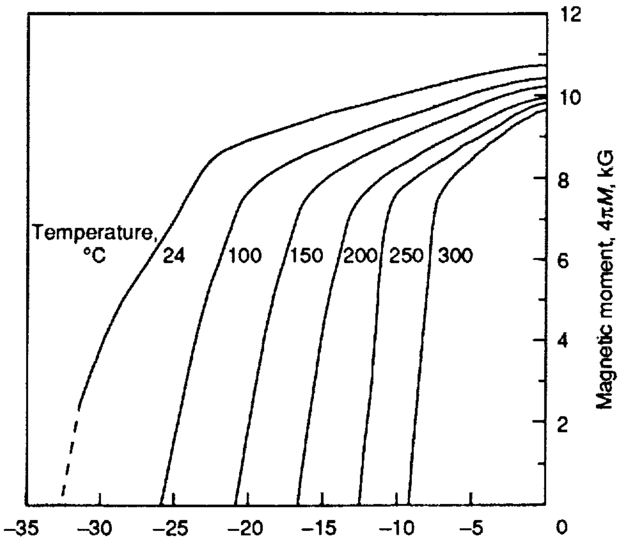

Figure 2.—Demagnetization characteristics of a commercial Sm_2Co_{17} magnet sample at selected temperatures to 300 °C. A pronounced *M–H* curve top droop was observed for all samples tested.

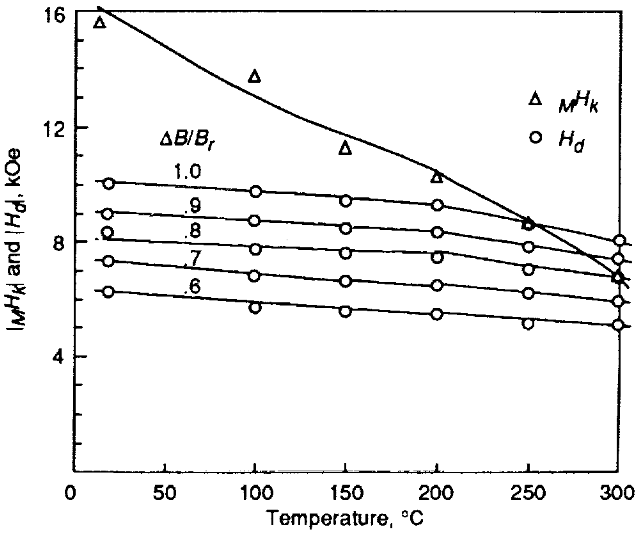

Figure 3.—Temperature variation of knee field $_MH_k$ and demagnetizing field H_d at selected induction swings derived from data in fig. 2. Benefits of the high room-temperature $_MH_k$ are quickly reduced by the high d_MH_k/dT.

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (*Leave blank*)

2. REPORT DATE

3. REPORT TYPE AND DATES COVERED
Technical Memorandum

4. TITLE AND SUBTITLE
Makeup and Uses of a Basic Magnet Laboratory for Characterizing
High-Temperature Permanent Magnets

5. FUNDING NUMBERS
WU-590-13-11

6. AUTHOR(S)
Janis M. Niedra and Gene E. Schwarze

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135-3191

8. PERFORMING ORGANIZATION REPORT NUMBER
E-6373

9. SPONSORING/MONITORING AGENCY NAMES(S) AND ADDRESS(ES)

National Aeronautics and Space Administration
Washington, D.C. 20546-0001

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
NASA TM-104508

11. SUPPLEMENTARY NOTES
Prepared for the First International High Temperature Electronics Conference cosponsored by Sandia National Laboratories, Wright Research and Development Center, and the University of New Mexico, Albuquerque, New Mexico, June 16-20, 1991. Janis M. Niedra, Sverdrup Technology, Inc., Lewis Research Center Group, 2001 Aerospace Parkway, Brook Park, Ohio 44142; Gene E. Schwarze, NASA Lewis Research Center. Responsible person, Gene E. Schwarze (216) 433-6117.

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Unclassified - Unlimited
Subject Category 33

12b. DISTRIBUTION CODE

13. ABSTRACT ***(Maximum 200 words)***

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14. SUBJECT TERMS
Permanent magnets; Rare-earth magnet; Samarium cobalt magnet; Demagnetization curve; High temperature; High coercivity; Magnet hysteresigraph

15. NUMBER OF PAGES

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT
Unclassified

18. SECURITY CLASSIFICATION OF THIS PAGE
Unclassified

19. SECURITY CLASSIFICATION OF ABSTRACT
Unclassified

20. LIMITATION OF ABSTRACT