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PHYSICAL PROPERTIES OF RARE EARTH-COBALT MAGNET MATERIALS

DAVID R. CHIPMAN and LAURENCE D. JENNINGS, Jr.
MATERIALS SCIENCES DIVISION

February 1972

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Technical Report by

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PHYSICAL PROPERTIES OF RARE EARTH-COBALT MAGNET MATERIALS

ABSTRACT

The magnetization of a number of commercial $SmCo_5$ magnets has been measured as a function of stress and temperature. These measurements are significant to present and proposed applications of the magnets and also to characterization and an understanding of the mechanisms involved. Application of a compressive stress of 10,000 psi, which is near the strength of the material, has a negligible effect at temperatures up to 215 C, at least. Heating the sample, on the other hand, induces not only the previously studied reversible and irreversible losses, but also time-dependent and permanent loss. The time-dependent losses indicate a range of relaxation times. The dependence of these relaxation times and of the permanent loss on external parameters and on sample history is complicated, and only preliminary characterization has been obtained to date, as illustrated by a number of examples.

FOREWORD

This report covers work done in the period July 1 to December 31, 1971, under the general title *Physical Properties of Rare Earth-Cobalt Magnet Materials*. The work is sponsored by the Advanced Research Projects Agency under ARPA Order No. 1914, Program Code No. ID10. The work was carried out at the Army Materials and Mechanics Research Center, Watertown, Massachusetts, 02172, by the principal investigators, D. R. Chipman and L. D. Jennings (Phone: 617 – 926-1900, Ext. 386 or 375).

CONTENTS

INTRODUCTION

Rare earth-cobalt (RE-C) intermetallic compounds offer enormous advantages over all other known materials for use as permanent magnets. These advantages have been discussed in a general way[1] and quite detailed characterizations have been made by the leading manufacturers of the present-day commercial magnets.[2,3] These characterizations have been directed primarily toward properties important to the predominant present usage: focusing magnets in traveling wave tubes (TWT). The RE-C magnets, however, display their greatest superiority over conventional magnets in dynamic applications, that is, under conditions of varying demagnetizing field. For example, a lightweight, high-speed, electrical power generator has been designed.[4] This generator has a design speed of 60,000 rpm and the corresponding compressive stress is about 10,000 psi. It is somewhat difficult to calculate the operating temperature with precision, but it will probably be above 200 C, and operation at 300 C or more would alleviate the cooling problem in similar applications.

We have made measurements of the loss of magnetization under stress and temperature conditions in the range mentioned above. The stress measurements have not been made previously. There have, on the other hand, been a large number of experiments at elevated temperature because the TWT operate hot. Anticipating our results, we find that there is negligible effect of stress at practicable levels. At temperatures near 200 C we find, in addition to the reversible and irreversible effects previously reported,[2-3] permanent and time-dependent effects, which have not been reported in closed pore RE-C sintered magnets. These latter represent losses of a few percent in the magnetization and thus are not terribly important from the technical point of view. It is probably for this reason that they have been overlooked in previous studies. On the other hand, these additional effects, particularly the time dependence, would be expected to give clues to the mechanism of the loss of magnetization. Since an understanding of this mechanism is perhaps the most important unknown in RE-C technology, we attach some significance to a study of these temperature-dependent effects. We report here some typical results obtained to date, but they should be considered very incomplete.

INSTRUMENTATION

In order to make measurements under varying conditions of applied magnetic field, stress, and temperature, we constructed an oscillating sample magnetometer.[4-6] The sample holder was designed to accommodate cylindrical specimens 0.02" in diameter and 0.10" high. Force could be applied to the sample through pressure-distributing pads which were compressed by tightening calibrated titanium bars. Our usual force was 314 pounds, giving a stress of 10,000 psi. The block which held the sample and the bars also contained two noninductively wound heaters on boron nitride forms. There was negligible coupling between the heaters and the pickup coils even though our temperature controller was suited only to an ac output. Temperature measurement and control was via a thermocouple imbedded in the block near the sample. The block-bar assembly was covered by a tetrafluoroethylene cover and oscillated vertically at 1 Hz, with an amplitude of 1.25". There were four pickup coils, each containing 250 turns with an average diameter of 0.80". The pairs were separated horizontally by 1.25" and vertically by 1.15". The axes of the coils, the magnetization of the sample, and the applied magnetic field were all horizontal and parallel. Because of the bulk of the pads, the bars, the tightening screws, and the insulating cover, the magnet gap was 1-5/8" and the maximum applied field available to us was 9 kilo-oersted. This bulk also limited our maximum heating rate to about 100 C/min. Therefore, when pressure was not required, we often removed the bars and replaced the pads with aluminum plugs. This arrangement produced a more uniform temperature distribution in the vicinity of the sample and also allowed a heating rate of about 300 C/min.

The output of the pickup coils was amplified and integrated using standard operational amplifier circuitry and then rectified using a diode and capacitor detector. The time constants were adjusted so that the rectified output was approximately equal to the peak-to-peak output of the integrator, which in turn is proportional to the magnetization of the sample. The value was read with a digital voltmeter. In actual fact, because of nonideality of the diodes, the apparatus was nonlinear, but suitable correction factors were determined and applied when necessary. For modest *changes* in magnetization, the correction was sensibly constant. The short-term precision of the apparatus was about 0.02% for typical samples magnetized near saturation, and about 0.1% over a long term.

Absolute calibration was made with the help of a nickel sample magnetized to saturation. Our configuration was such that about 30% of the induced voltage arose from image poles. Thus the calibration constant was about 30% less when the magnetometer was removed from the magnet. We did, in fact, often use the magnetometer removed from the magnet when we were making open circuit measurements. Because of these complications, the absolute accuracy of the calibration was about 1%, which was more than adequate for the intended use of the results.

SAMPLES

We had at our disposal three samples from each of three different lots from two different manufacturers. We designate samples by a letter indicating the lot and a number to distinguish samples within a lot. Lots A, B, and C were supplied by the General Electric Co., which makes use of a process utilizing an additive which liquefies at the sintering temperature. Lots S, T, and U were supplied by Raytheon Co., which utilizes a single, multiphase starting material. All samples were stated to be representative of current (August 1971) production, except for lot A which was specially processed; all are nominally $SmCo_5$.

In general, each magnet was magnetized at room temperature before each run. For most of our work, this was carried out with a 100 kilo-oersted pulsed field. In our preliminary measurements, however, we had only a 30 kilo-oersted field from an iron core magnet at our disposal. Either field fully magnetized the General Electric samples, but the 30 kilo-oersted field was about 1-1/2% less effective for the Raytheon samples under most conditions.

STRESSED MEASUREMENTS

As stated in the Introduction, the behavior of the magnets under stress was completely uninvestigated before our work. Therefore, because of the importance of such behavior in applications, we gave our primary attention to it. Furthermore, the expected operating conditions of the magnet in the MERDC generator design are: temperature, 215 C; demagnetizing field, 4 kilo-oersted, and stresses ranging up to 10,000 psi. We considered these values to be not only representative of design requirements, but they are also both convenient and reasonable. Irreversible losses are appreciable but not drastic at 215 C. A demagnetizing field of 4 kilo-oersted is near the point of maximum energy product and also is sufficiently large to give significant irreversible effects.[3] Since the average demagnetizing factor (in rationalized units) is 0.475 for our samples, we also find that the open circuit demagnetizing field is about 4 kilo-oersted for a typical case. We therefore have made most of our measurements in the open circuit configuration. Lastly, 10,000 psi is near the strength level quoted by the manufacturers. In fact, we found that all our samples were able to withstand a single compressive stress of this level, but one sample failed after 100 cycles at this level. Thus we felt that 10,000 psi was near the limit of the stress that we could safely use without losing samples.

Runs which we consider typical of our most stringent tests for a stress effect are shown in Figure 1. The run on the virgin sample is shown in Figure 1a, where magnetization is plotted versus time at temperature. After 8 minutes at 215 C, the sample was heated to 225 C with the hope that it would be quickly stabilized. The temperature was then restored to 215 C, and the magnetization was observed with the applied stress alternately zero and P_S = 10,000 psi. After the run depicted in Figure 1a, the sample was cooled to room temperature and remagnetized. A similar run was carried out as shown in Figure 1b. This time, however, the sample was maintained at temperature for 15 hours, after which the results shown in Figure 1c were obtained.

There are a number of considerations which are important in correctly interpreting the runs of Figure 1. Most important from a technical point of view is the fact that temperature control between the $P = 0$ case and the $P = P_S$ case is extremely difficult. The reason for this is that the thermal impedances between the heater, the thermocouple, the titanium bars, and the sample change when the stress is applied. This change gives rise to both transient and steady state effects. These effects could have been mitigated somewhat with a more massive, better insulated apparatus, but such an apparatus would have been inconveniently bulky. Instead, we made measurements of the temperature distribution for $P = 0$ and $P = P_S$ and then made corrections to the P_S data. These corrections are equivalent to about 0.15% in the magnetization, and have been applied to the data of Figure 1. These corrections are, however, valid only in the steady state. Under transient conditions, which lasted about 7 minutes, the situation depended on the way the temperature controller was utilized. Examples which clearly arise from this effect are the anomalous changes in slope in Figure 1a, the effects between 50 and 75 minutes in Figure 1b, and the two P_S cases in Figure 1c where the control was deliberately offset a small but easily observable amount in each case during the actual stress change. Thus in cases such as those shown in Figure 1a where there is a relatively large time dependence, it was not possible to make a very precise measurement of the stress effect. After the sample had stabilized, higher precision was possible, limited only by the knowledge of the steady state correction.

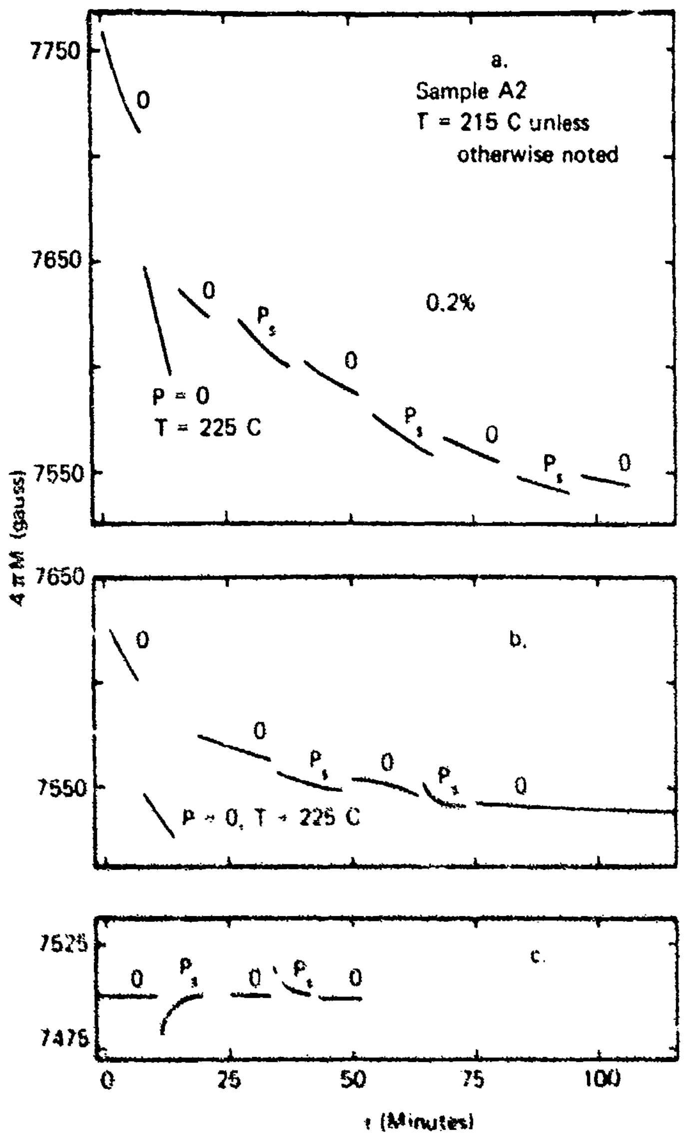

Figure 1. A typical run to determine the effects of stress on a RE-C magnet. P_s refers to the standard uniaxial stress of 10,000 psi; 0 to an unstressed condition. See text for a discussion of the various apparent anomalies.

Runs similar to those shown in Figure 1 were made on at least one sample from each lot. We also made similar comparisons at room temperature and after cycling to P_s 100 times. In every case our results are consistent with the hypothesis that there is no effect of stress within the following limits:

At room temperature, application of P_s has less than 0.02% effect on the magnetization.

Cycling to P_s 100 times has less than 0.1% effect.

Near 215 C, application of P_s has less than 0.05% effect on a well stabilized magnet.

Near 215 C, application of P_s has less than 0.2% effect on a poorly stabilized magnet. Our measurements seem to show a small effect, on the average, but this amount is barely within our precision under these conditions.