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On the standard measuring method for magnetic properties of permanent magnet materials with very high coercivity

Yasuaki Nakagawa[a] and Hiroaki Kato[b]

[a]Department of Electronics, Tohoku Institute of Technology, Yagiyama, Taihaku-ku, Sendai 982, Japan

[b]Institute for Materials Research, Tohoku University, Katahira, Aoba-ku, Sendai 980, Japan

A saturated hysteresis loop for permanent magnet materials with very high coercivity can be measured only in an open magnetic circuit, where a correction for the demagnetizing field becomes an important problem except for very thin samples. We have measured the magnetization of some magnetically hard materials of various shape. Even the intrinsic coercivity seems to depend on the dimension ratio of the samples in some cases. Standard specifications of rare earth magnets are discussed on the basis of these measurements.

1. INTRODUCTION

Standard specifications and measuring methods for permanent magnet materials have been described in publications of International Electrotechnical Commission (IEC) [1-3]. Although the IEC standard deals with high coercivity materials such as samarium cobalt, the use of a closed magnetic circuit consisting of an iron-core electromagnet is still recommended in spite of the fact that the full saturation of magnetization requires much higher magnetic fields [2].

This problem has become more serious since the discovery of rare earth iron boron magnets having a coercivity of more than 2 T which corresponds to the saturation flux density of pure iron or iron-cobalt alloys. Although higher magnetic fields can be produced by a superconducting coil or a high-power resistive coil, it is difficult to realize the closed magnetic circuit. Thus the measurement of the magnetization or the magnetic polarization should be made in an open magnetic circuit by using a vibrating sample magnetometer, a sample extraction magnetometer or a magnetic balance. The magnetic field in the sample is modified by the demagnetizing field due to a surface magnetic charge which depends on the shape of the sample.

The open magnetic circuit method has been described in the IEC publication only for the determination of the intrinsic coercivity, which is thought to be unaffected by the demagnetizing field [3]. In the present work we have measured magnetization curves for various samples of commercial rare earth magnets in the open magnetic circuit. The standard measuring method is discussed together with the standard characteristics of these materials.

2. EXPERIMENTAL

The commercial rare earth magnets examined in the present work are as follows: sintered Nd-Fe-B magnets, sintered Sm-Co magnets, bonded Nd-Fe-B magnets, and bonded Sm-Co magnets. The samples are circular cylinders of 4 mm in length with various diameters of 1, 2, 3 and 4 mm.

Steady magnetic fields up to 23 T are produced by a hybrid magnet [4] consisting of an outer superconducting coil of 8 T and an inner high-power resistive coil of 15 T. The magnetization is measured by a sample extraction method [5]; the voltage induced by the extraction of the magnetized sample is detected by a pick-up coil. Four specimens were set simultaneously in the magnetizing coil and the extraction was made successively during a slow sweep of the field. The field should be so uniform that the difference in field strength between the end positions of the sample extraction is negligibly small. This is especially important for the measurement of the magnetization curve in the second quadrant which exhibits a very steep change near the coercivity point. Therefore, after the magnetization was saturated in the first quadrant by the hybrid magnet, only the superconducting coil was energized for the measurement in the second quadrant to give more uniform fields. Experimental arrangements are shown in fig. 1 schematically.

The effective field H_{eff} in the sample is derived from the applied field H_{app} by

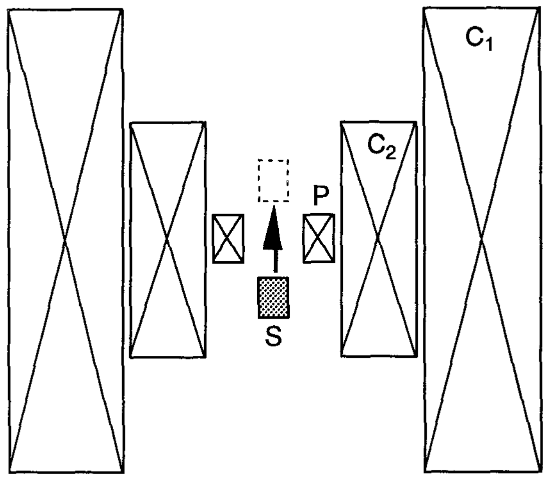

Figure 1. Hybrid magnet and sample extraction magnetometer. C_1: superconducting coil. C_2: high-power resistive coil. P: pick-up coil. S: sample.

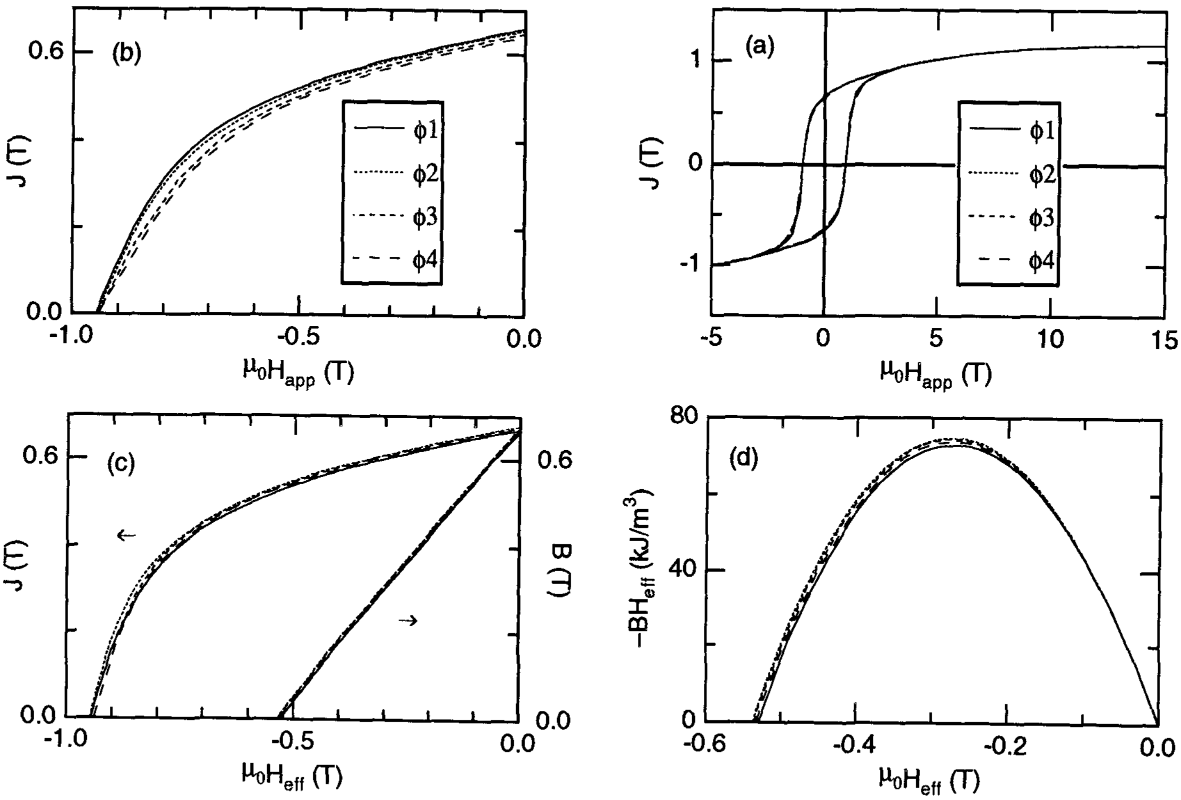

Figure 2. Magnetizations of bonded Nd-Fe-B magnet (isotropic). Samples are circular cylinders of 4 mm in length and 1, 2, 3 and 4 mm in diameter.

$$\mu_0 H_{eff} = \mu_0 H_{app} - NJ, \tag{1}$$

where J is the magnetic polarization and N the demagnetizing factor. The fields are multiplied by μ_0 to be expressed by the same units as the magnetic polarization. Numerical values of N were estimated from the measurement on pure nickel samples of the same dimensions. It was assumed that the nickel samples were magnetically soft enough so that the initial slope of the magnetization curve became infinitely large after the correction for the demagnetizing field. Values of J were also calibrated by the measurement of the saturation value for the pure nickel samples.

3. RESULTS

Figure 2 shows experimental results on the bonded Nd-Fe-B magnet with an isotropic magnetization. The magnetic polarization J is plotted as a function of the applied field $\mu_0 H_{app}$ from +15 T to –5 T in fig. 2(a). Although the saturation of J requires the field of 10 T, the hysteresis appears only below 3 T. Figure 2(b) shows the second quadrant in an enlarged scale, clarifying differences among the four samples. The intrinsic coercivity does not depend on the sample shape since the demagnetizing field vanishes at this point. The larger the diameter of the cylindrical sample, the smaller becomes the apparent value of the remanence. These curves become almost congruent after the correction for the demagnetizing field, as shown in fig. 2(c). The real value of the remanence is evidently larger than a half of the saturation magnetization expected from the isotropic magnetization. The magnetic flux density B given by

$$B = \mu_0 H_{eff} + J \tag{2}$$

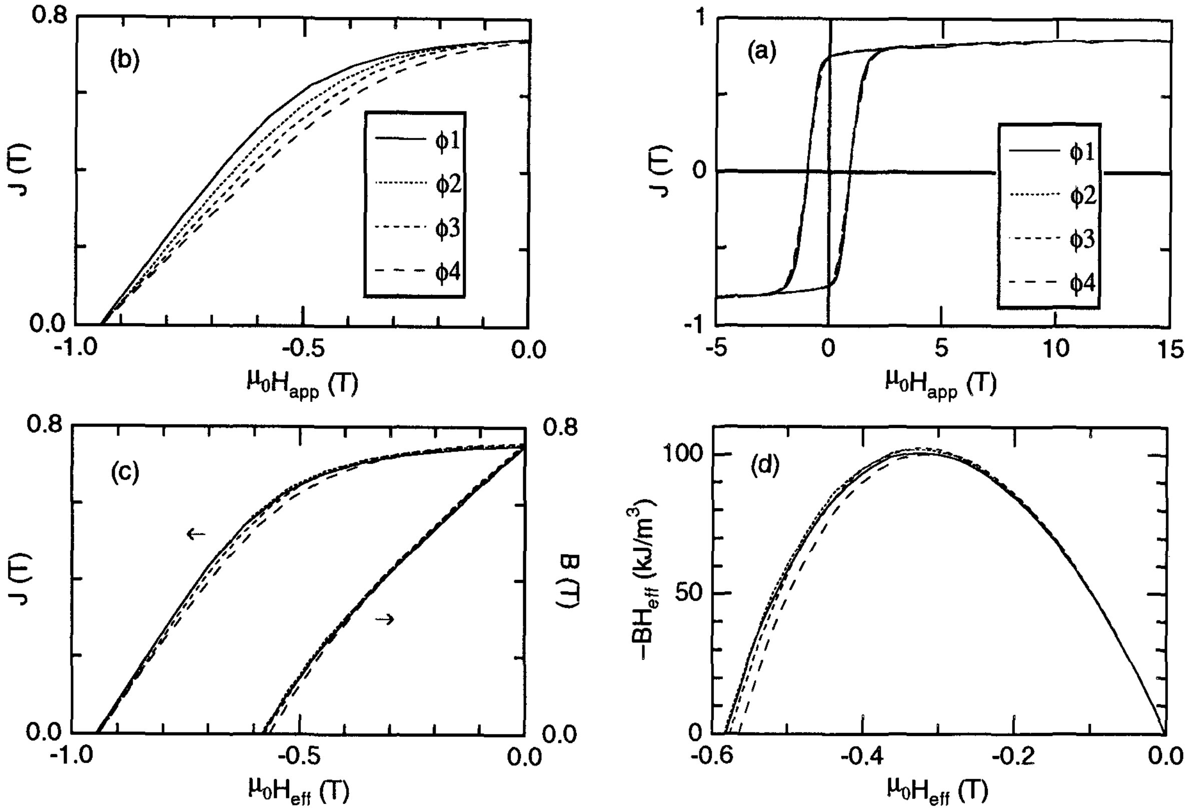

Figure 3. Magnetizations of bonded Sm-Co magnet (anisotropic). Samples are circular cylinders of 4 mm in length and 1, 2, 3 and 4 mm in diameter.

is also plotted in this figure. The coercivity of B is much smaller than the intrinsic coercivity (of J). The energy product $-BH_{eff}$ in the second quadrant is plotted in fig. 2(d). The maximum point on the curve gives the so-called $(BH)_{max}$ value. There are little differences in $(BH)_{max}$ among the four samples.

Similar results are shown in fig. 3 for the bonded Sm-Co magnet with an anisotropic magnetization due to the magnetic alignment. The field was applied along the direction of easy magnetization, resulting in easier approach to saturation, as shown in fig. 3(a). Different curves for the different samples in fig. 3(b) are in close resemblance after the correction for the demagnetizing field in fig. 3(c), similarly to those in fig. 2. Although an appreciable discrepancy is still found in the enlarged figures, we may conclude that the correction is satisfactory for both isotropic and anisotropic bonded magnets.

In the case of the sintered Sm-Co magnet shown in fig. 4, on the other hand, even the intrinsic coercivity depends on the diameter of samples. Figure 4(c) exhibits that the curves after the correction for the demagnetizing field are quite different from each other. Also the $(BH)_{max}$ value depends on the sample shape, as shown in fig. 4(d). The smaller the diameter of the cylindrical sample, the larger become both the intrinsic coercivity and the $(BH)_{max}$ value. It seems that the results for the sample of 1 mm in diameter represents the most reliable characteristics of this material since the correction is smallest for this sample.

Figure 5 shows the magnetizations of the sintered Nd-Fe-B magnet with very large coercivity. The anomalous decrease in magnetization is found in the second quadrant near the remanence point. This may be due to the deterioration of the surface region of the test samples [6]. Thus the smallest sample exhib-

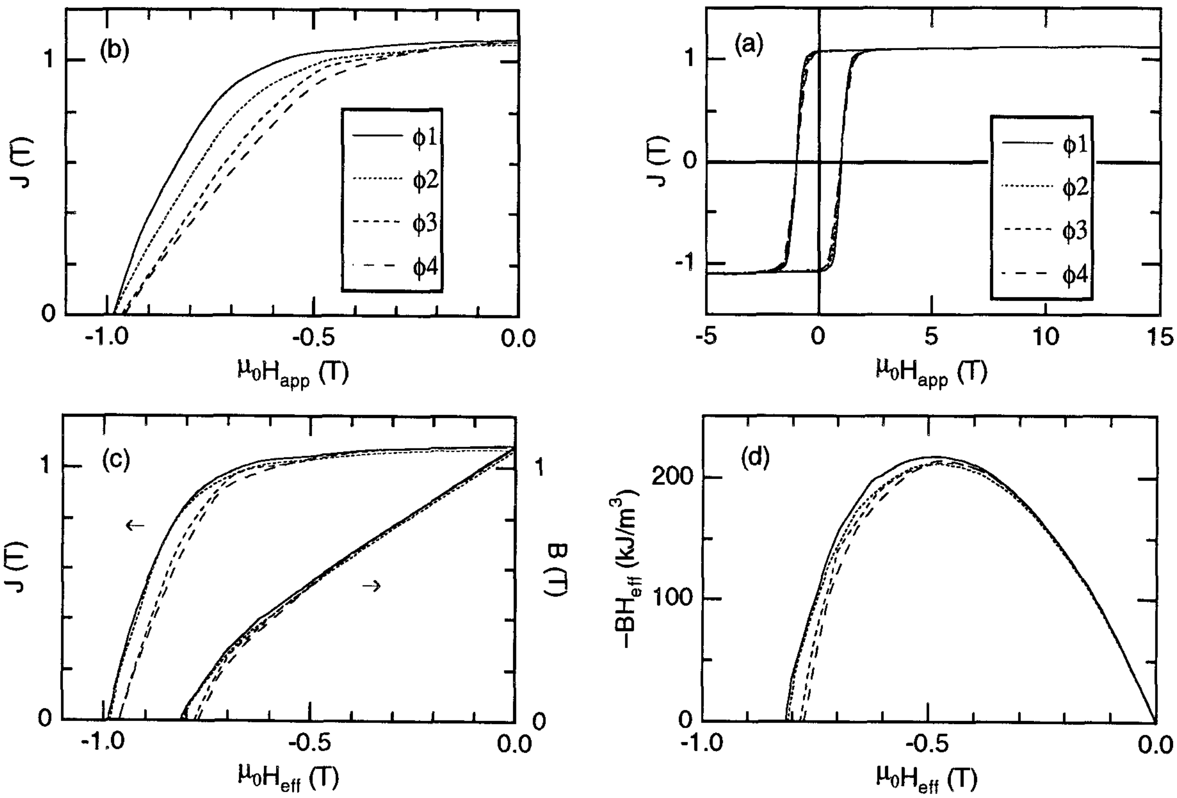

Figure 4. Magnetizations of sintered Sm-Co magnet. Samples are circular cylinders of 4 mm in length and 1, 2, 3 and 4 mm in diameter.

its the most remarkable decrease. Taking also the above result on the sintered Sm-Co magnet into consideration, we may conclude that neither the large-diameter sample nor the small-diameter sample of the sintered Nd-Fe-B magnet gives a satisfactory result. It is expected, however, that the sintered Nd-Fe-B magnet will behave similarly to the sintered Sm-Co magnet if the surface deterioration can be suppressed.

4. DISCUSSION

The open magnetic circuit method is proved to be successful to characterize the bonded magnet materials since the correction for the demagnetizing field gives the reasonable result. We have used the demagnetizing factors estimated from the measurement on pure nickel samples. It is well known, however, that the demagnetizing field in a cylindrical sample is not uniform and depends on the permeability of the material. Thus the demagnetizing factors used here are not fully justified. Although a detailed calculation of demagnetizing factors of circular cylinders has been made in the case where the permeability is constant [7], this is inapplicable to the magnetization curve in the second quadrant. At the present stage, we are merely satisfied by the fact that a unique magnetization curve has been obtained for the bonded magnet samples.

On the other hand, the measurements in the open magnetic circuit for the sintered Sm-Co and Nd-Fe-B magnets are not satisfactory. It should be emphasized, however, that the closed magnetic circuit method would never be applicable to these materials, of which the intrinsic coercivity (multiplied by μ_0) is comparable to the saturation flux density of iron or iron-cobalt alloys. It has been recommended in the IEC publication [2] that the flux density

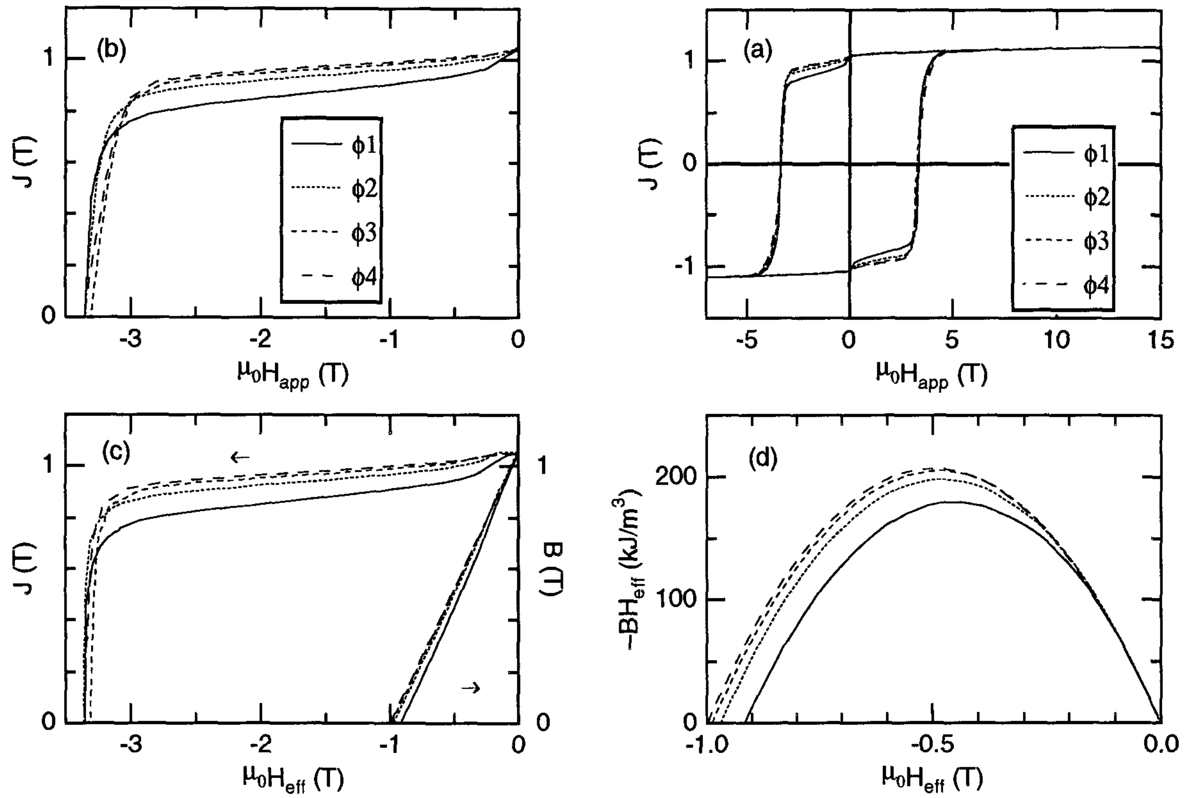

Figure 5. Magnetizations of sintered Nd-Fe-B magnet. Samples are circular cylinders of 4 mm in length and 1, 2, 3 and 4 mm in diameter.

in the closed magnetic circuit should be less than 1 T in iron and less than 1.2 T in iron-cobalt alloys.

It has been hoped that the standard specifications of the magnetic materials are determined to be independent of the shape of the test sample. This may be possible if the closed magnetic circuit is usable, i.e. the coercivity of the sample is sufficiently less than 1.2 T. The materials having higher coercivity should be examined by the open magnetic circuit method, which inevitably depends on the correction for the demagnetizing field.

It is unlikely that the non-uniformity of the demagnetizing field in the cylindrical sample plays an important role. A more serious problem is the non-uniformity due to a coarse magnetic domain structure, which may be realized in the sintered magnet sample having the magnetization in the second quadrant. An apparent dependence of the intrinsic coercivity on the sample shape is thought to be due to the difference in the domain structure. This may occur even in a spherical or an ellipsoidal sample, in which the demagnetizing factors for the uniform magnetization can be determined by the macroscopic electromagnetic theory. The demagnetizing field is not uniform, however, even in a macroscopic scale, if the spherical or ellipsoidal sample has the coarse domain structure. The bonded magnets without the coarse domain structure give the satisfactory result since the magnetization is macroscopically uniform except for the non-uniformity due to the cylindrical shape. Thus the correction for the average demagnetizing field is useful in this case.

The surface deterioration in the present samples of the sintered Nd-Fe-B magnet is a completely different problem. This may be solved by an adequate treatment of the samples, and then we may encounter the same problem as in the sintered Sm-Co magnet. In general it is difficult to give the standard specifications by the measurement in the open magnetic circuit, even when the spherical or ellipsoidal sample is used. Thus we shall be satisfied by giving more practical specifications which may depend on the sample shape. For this purpose, the elaborate measurement using the hybrid magnet is unnecessary. Conventional measurements in high magnetic fields can be made by using a pulsed field magnet or a small superconducting magnet.

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