Designation: A 977/A 977M – 02

Standard Test Method for Magnetic Properties of High-Coercivity Permanent Magnet Materials Using Hysteresigraphs[1]

This standard is issued under the fixed designation A 977/A 977M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes how to determine the magnetic characteristics of magnetically hard materials (permanent magnets), particularly their initial magnetization, demagnetization, and recoil curves and such quantities as the residual induction, coercive fields, knee field, energy products, and recoil permeability. This test method is suitable for all materials processed into bulk magnets by any common fabrication technique (casting, sintering, rolling, molding, and so forth), but not for thin films or for magnets that are very small or of unusual shape. Uniformity of composition, structure, and properties throughout the magnet volume is necessary to obtain repeatable results. Particular attention is paid to the problems posed by modern materials combining very high coercivity with high saturation induction, such as the rare-earth magnets, for which older test methods (see Test Method A 341) are unsuitable. An applicable international standard is IEC Publication 404-5.

1.2 The magnetic system (circuit) in a device or machine generally comprises flux-conducting and nonmagnetic structural members with air gaps in addition to the permanent magnet. The system behavior depends on properties and geometry of all these components and on the temperature. The tests described here measure only the properties of the permanent magnet material. The basic test method incorporates the magnetic specimen in a magnetic circuit with a closed flux path. Test methods using ring samples or frames composed entirely of the magnetic material to be characterized, as commonly used for magnetically soft materials, are not applicable to permanent magnets.

1.3 This test method shall be used in conjunction with Practice A 34/A 34M.

1.4 The values and equations stated in customary (cgs-emu or inch-pound) or SI units are to be regarded separately as standard. Within this test method, SI units are shown in brackets except for the sections concerning calculations where there are separate sections for the respective unit systems. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with this test method.

1.5 The names and symbols of magnetic quantities used in this test method, summarized in Table 1, are those currently preferred by U.S. industry.

1.6 This test method is useful for magnet materials having H_{ci} values between about 100 Oe and 35 kOe [8 kA/m and 2.8 MA/m], and B_r values in the approximate range from 500 G to 20 kG [50 mT to 2 T]. High-coercivity rare-earth magnet test specimens may require much higher magnetizing fields than iron-core electromagnets can produce. Such samples must be premagnetized externally and transferred into the measuring yoke. Typical values of the magnetizing fields, H_{mag}, required for saturating magnet materials are shown in Table 1.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

A 34/A 34M Practice for Procurement Testing and Sampling of Magnetic Materials[2]

A 340 Terminology of Symbols and Definitions Relating to Magnetic Testing[2]

A 341/A 341M Test Method for Direct Current Magnetic Properties Using dc Permeameters and the Ballistic Test Methods[2]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[3]

2.2 *Magnetic Materials Procedure Association Standard:*

MMPA No. 0100–96 Standard Specifications for Permanent Magnet Materials[4]

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[1] This test method is under the jurisdiction of ASTM Committee A06 on Magnetic Properties and is the direct responsibility of Subcommittee A06.01 on Test Methods.

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[2] *Annual Book of ASTM Standards*, Vol 03.04.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Available from Magnetic Materials Producers Association, 8 S. Michigan Ave., Suite 1000, Chicago, IL 60603.

TABLE 1 Symbols, Quantities, and Units

NOTE 1—IEC nomenclature calls B_r “remanence,” when B_r represents the B at $H = 0$ of the outermost hysteresis loop, and it calls B_r “remanent magnetic induction” for B at $H = 0$ at smaller loops.

Symbol	Quantity	SI Unit	Customary cgs-emu
A_t	Cross section of search coil	[m^2]	cm^2
B_d	Magnetic induction at BH_{max}	[T]	G
B_{rec}	Magnetic induction at low point of recoil loop	[T]	G
B_r	Magnetic induction at remanence	[T]	G
d_l	Diameter of pole piece	[m]	cm
d_2	Diameter of homogeneous field	[m]	cm
H_d	Magnetic field strength at BH_{max}	[A/m]	Oe
H_p	Magnetic field strength at low point of recoil loop	[A/m]	Oe
l	Distance between pole faces	[m]	cm
l_r	Length of test sample	[m]	cm
N	Number of turns of test coil		
e	Voltage induced in test coil	V	V
d	Total air gap between test sample and pole faces	[m]	cm
μ_0	A constant with value $\mu_0 = 4\pi\ 10^{-7}$ H/m		
μ_{rec}	Recoil permability		

2.3 *International Electrotechnical Commission Document:* Publication 404-5 Magnetic Materials – Part 5: Permanent Magnet (Magnetically Hard) Materials – Methods of Measurement of Magnetic Properties[5]

3. Terminology

3.1 Basic magnetic units are defined in Terminology A 340 and MMPA Standard No. 0100–96. Additional definitions with symbols and units are given in Table 1 and Figs. 1-3 of this test method.

4. Significance and Use

4.1 This test method is suitable for magnet specification, acceptance, service evaluation, quality control in magnet production, research and development, and design.

4.2 When a test specimen is cut or fabricated from a larger magnet, the magnetic properties measured on it are not necessarily exactly those of the original sample, even if the material is in the same condition. In such instances, the test results must be viewed in context of part performance history.

4.3 Tests performed in general conformity to this test method and even on the same specimen, but using different test systems, may not yield identical results. The main source of discrepancies are variations between the different test systems in the geometry of the region surrounding the sample, such as, size and shape of the electromagnet pole caps (see Annex A1 and Appendix X1), air gaps at the specimen end faces, and especially the size and location of the measuring devices for H and B or for their corresponding flux values (Hall-effect probes, inductive sensing coils). Also important is the method of B calibration, for example, a volt-second calibration of the fluxmeter alone versus an overall system calibration using a physical reference sample. The method of B and H sensing should be indicated in test reports (see Section 9).

5. Measuring Methods and Apparatus

5.1 *Measuring Flux and Induction (Flux Density)*:

5.1.1 In the preferred B-measuring method, the total flux is measured with a sensing coil (search coil) that surrounds the test specimen and is wound as closely as possible to the specimen surface. Its winding length should be no more than a third of the specimen length, preferably less than one fifth, and must be centered on the specimen. The leads shall be twisted tightly. As the flux changes in response to sweeping the applied field, H, the total flux is measured by taking the time integral of the voltage induced in this coil. This measurement is taken with a fluxmeter. Modern hysteresigraphs use electronic integrating fluxmeters that allow convenient continuous integration and direct graphic recording of magnetization curves. If the signal is large enough, high-speed voltage sampling at the coil and digital integration is also possible.

5.1.2 The magnetic induction, B, is determined by dividing the total flux by the area-turns product, NA, of the B-sensing coil. For permanent magnets in general, and especially for high-coercivity materials, an air-flux correction is required (see 5.3 and 5.4).

5.1.3 The total error of measuring B shall be not greater than ±2 %.

5.1.4 The change of magnetic induction, $\Delta B = B_2 - B_1$, in the time interval between the times t_1 and t_2 is given as follows:

$$\Delta B = (10^8/AN) \int_{t_1}^{t_2} e\,dt \text{ (customary units)} \tag{1}$$

$$\Delta B = (1/AN) \int_{t_1}^{t_2} e\,dt \text{ (SI units)} \tag{2}$$

where:

B = magnetic induction, G [T];
A = cross-sectional area of the test specimen, cm^2 [m^2];
N = number of turns on the B-sensing coil;
e = voltage induced in the coil, V;
t = time, s; and
$\int_{t_1}^{t_2} e\,dt$ = voltage integral = flux, V-s [Weber].

5.1.5 The change in the magnetic induction shall be corrected to take into account the air flux outside the test specimen that is linked by the sensing coil. The corrected change, B_{corr}, is given as follows:

$$\Delta B_{corr} = (10^8/AN) \int_{t_1}^{t_2} e\,dt - \Delta H\,(A_t - A)/A \text{ (customary units)} \tag{3}$$

$$\Delta B_{corr} = (1/AN) \int_{t_1}^{t_2} e\,dt - \mu_0\,\Delta H\ (A_t - A)/A \text{ (SI units)} \tag{4}$$

where:

A = average cross-sectional area of the sensing coil, cm^2 [m^2];
ΔH = change in field from t_1 until t_2, Oe [A/m]; and
μ_0 = magnetic constant [$4\pi\ 10^{-7}$ H/m].

5.2 *Determining Intrinsic Induction*:

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[5] Available from International Electrotechnical Commission (IEC), 3 rue de Varembé, P.O. Box 131, CH-1211, Geneva 20, Switzerland.

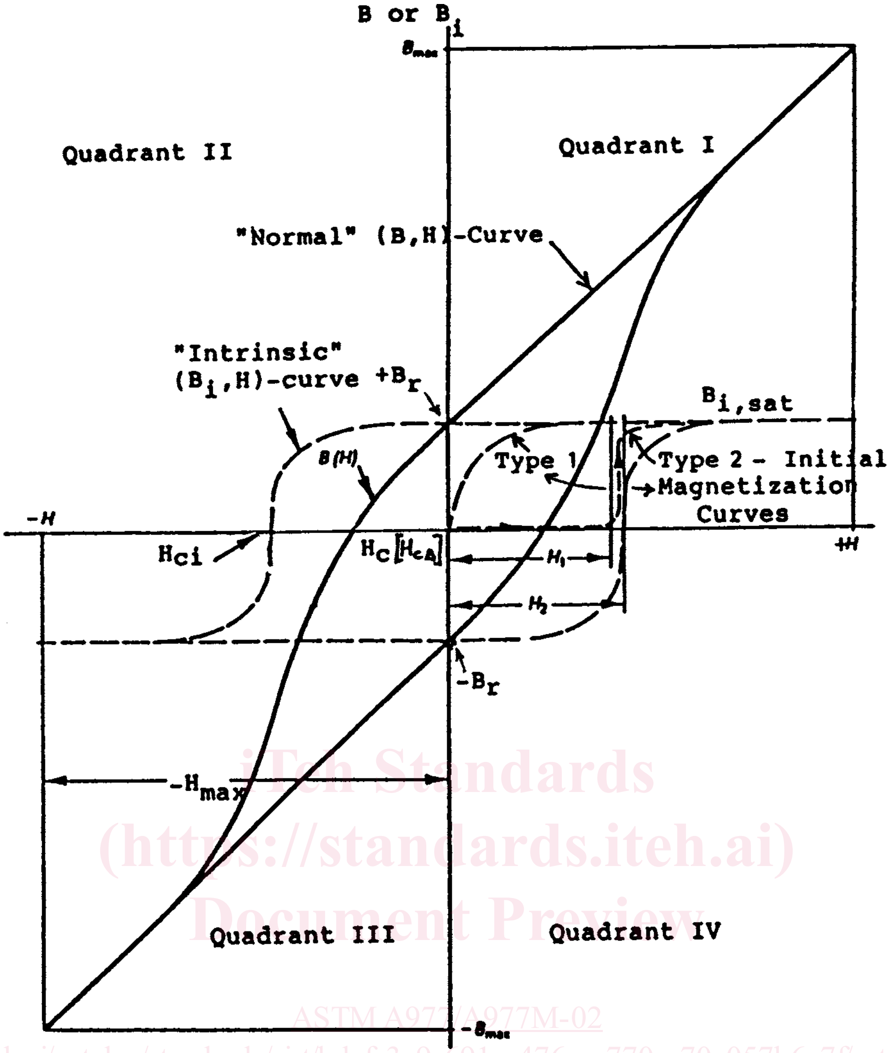

FIG. 1 Normal and Intrinsic Hysteresis Loops and Initial Magnetization Curves for Permanent Magnet Materials Illustrating Two Extremes of Virgin Sample Behavior

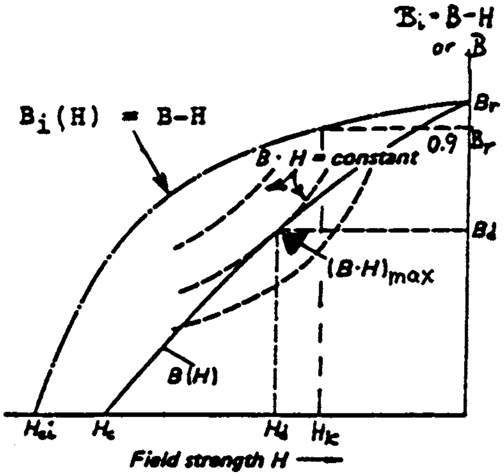

FIG. 2 Normal and Intrinsic Demagnetization Curves with Symbols for Special Points of Interest and Definition of Salient Properties. Illustration of Maximum Energy Product, Coercive Fields, and Definition of Knee Field

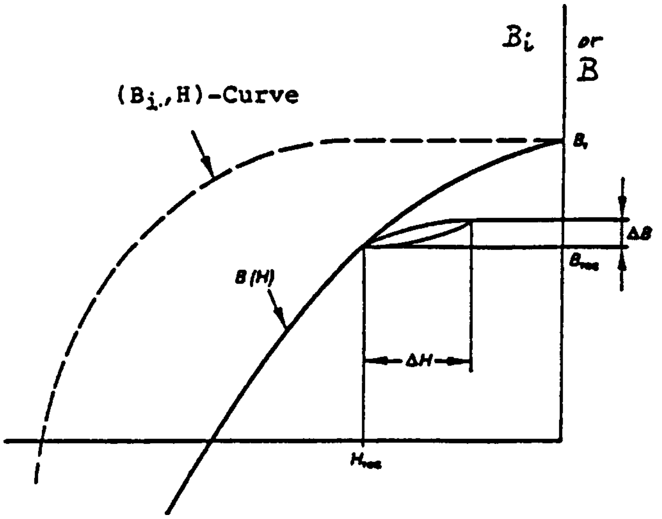

FIG. 3 Normal and Intrinsic Demagnetization Curves with Symbols for Special Points of Interest and Definition of Salient Properties. Illustration of Recoil Loop. Recoil Permeability is Defined as $\mu_{rec}\Delta B/\Delta H$

5.2.1 For high-coercivity magnets, it is more convenient to sense directly an electrical signal proportional to the intrinsic induction, derive the average B_i by dividing this flux by the area-turns product of the surrounding B coil, and to plot B_i versus H as the primary demagnetization curve. B then is obtained by mathematical or electronic addition of H to B.

5.2.2 The change of intrinsic induction in the test specimen can be determined by integrating the voltage induced in a device comprising two sensing coils, both subject to the same applied field H, where the test specimen is contained in only one of the coils (Coil 1). If each individual coil has the same area-turns product, and if the windings are connected electrically in opposition, the signal induced by the flux linking Coil 2 (not containing the specimen) will compensate for the output of Coil 1 except for B_i within the test specimen. The change of intrinsic induction in the specimen then is given as follows:

$$\Delta B_i = (10^8/AN) \int_{t_1}^{t_2} e\,dt \text{ (customary units)} \tag{5}$$

$$\Delta B_i = (1/AN) \int_{t_1}^{t_2} e\,dt \text{ (SI units)} \tag{6}$$

where:

B_i = intrinsic induction, G [T];
A = cross section of the test specimen, cm^2 [m^2]; and
N = number of turns on Coil 1 containing the test specimen.

5.2.3 The two-sensing-coil device shall lie totally within the homogeneous field defined by Eq A1.1 and Eq A1.2. Test specimens of lower-coercivity magnets having a range of cross-sectional areas and shapes can then be measured with the same coil device. An arrangement of side-by-side coils of equal size is useful. Serious errors, however, are incurred when measuring B_i this way on high-B_r or high/coercivity magnets, or both, at applied fields of about 10 kOe or more. The errors are most severe for test specimens of short pole-to-pole length. Local pole-piece saturation causes strong field inhomogeneities. The specimen then must fill the cross section of Coil 1, and Coil 2 must be a thin and flat coil, or a coaxial annular coil, either centered on the specimen or in close proximity to its surface (see 5.3).

5.2.4 The total error of measuring B_i shall be not greater than ±2 %.

5.3 *Measuring the Magnetic Field Strength*:

5.3.1 For correct magnetization curves, one should know the magnetic field strength, H, inside the test specimen, averaged over the specimen volume if H is not uniform. But this inner field cannot be measured. At the surface of the test specimen, H is equal to the local field strength just inside the specimen in those locations (and only there) where the H vector is parallel to the side surface of the specimen. Therefore, a magnetic field strength sensor of small dimensions relative to the specimen is placed near the specimen surface and symmetrical with respect to the end faces, covering the shortest possible center portion of the specimen length. It shall be so oriented that it correctly measures the tangential field component.

5.3.2 To determine the magnetic field strength, a flat surface coil, a tightly fitted annular coil, a magnetic potentiometer, or a Hall probe is used together with suitable instruments. The dimensions of the magnetic field sensor and its location shall be such that it is within an area of limited diameter around the test specimen (see Annex A1).

5.3.3 The provisions of 5.3.2 are adequate for measurements on magnets having low-to-moderate intrinsic coercivity, such as Alnico and bonded ferrites. For high-coercivity, dense ferrites and especially for most rare earth-transition metal materials, it is essential for accurate measurement to use thin flat or radially thin annular H-sensing coils of short length (<1/5 to 1/3 of the specimen length), centered on the specimen and placed as close as possible to the specimen surface.

5.3.4 The same considerations apply to the H-flux compensation coil used in B_i measurements (see 5.2.3.) When pole saturation can occur, Coil 2 also shall be a thin conforming flat surface coil for rectangular specimen shapes or a thin annular coil closely surrounding a cyclindrical specimen, and the specimen essentially shall fill the open cross-sectional area of the B–sensing Coil 1.

5.3.5 To reduce other measurement errors, the air gaps between the flat ends of the test specimen and the pole pieces shall be kept small, typically in the range 0.001 to 0.002 in. [0.025 to 0.050 mm] (see Fig. 4).

5.3.6 The magnetic field strength measuring system shall be calibrated. Any temperature dependence of the measuring instruments, (for example, Hall probes), must be taken into account. The total error of measuring H shall be not greater than ±2 %.

NOTE 1—The end faces of the test specimen should be in intimate contact with the pole faces. There are always unavoidable small air gaps as a result of surface roughness, poor parallelism of sample or pole faces, or intentional shimming to protect delicate specimens from deformation or crushing. These cause additional errors in the magnetic field strength measurement and indirectly in the B_i measurements through air flux compensation errors, even in the low H region. The maximum error in the field strength measurement, as a result of two symmetric gaps of length d (see Fig. 3) is approximately:

$$\Delta H/H = 2\,B\,d\,/\,l_r\,H \text{ (customary units)} \tag{7}$$

$$\Delta H/H = 2\,B\,d\,/\,\mu_0\,l_r\,H \text{ (SI units)} \tag{8}$$

To keep the error 100 $\Delta H/H < 1$ % in the region of the $(BH)_{max}$ point, the gap thickness should be kept below the following values:

$d = 0.00025\,l_r$ for Alnico magnets,
$d = 0.005\,l_r$ for hard ferrite magnets, and
$d = 0.003\,l_r$ for rare-earth magnets.

5.4 *Plotting Magnetization and Demagnetization Curves*:

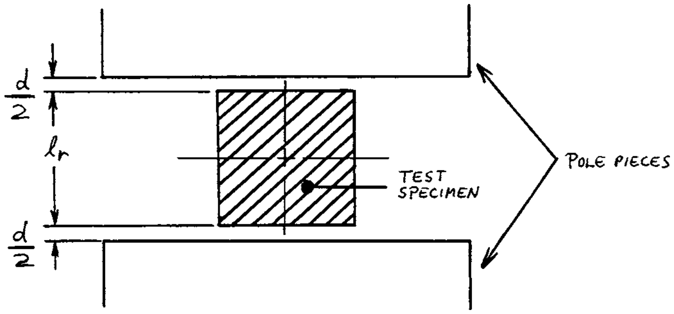

FIG. 4 Illustration Regarding the Influence of Air Gaps at the End Faces of the Test Specimen

5.4.1 Plotting of B_i, H curves or B, H curves is accomplished by combining one of the methods for magnetic field strength measurement from 5.3 with a B_i-measuring method from 5.2 or a B-measuring method from 5.1. A schematic for a typical hysteresigraph system is shown in Fig. 5.

5.4.2 *Continuous Plotting of Magnetization Curves*—Modern electronic integrators used in conjunction with inductive sensors for B_i or B, and in some instruments also for H, allow the continuous recording of magnetization, demagnetization, or recoil curves. A wide range of field sweep rates is possible. In the simplest but least desirable case, the exciting current of the electromagnet may be varied linearly, or the field sweep rate may be held constant. Even better it may be controlled with feedback from the measuring circuit for the (intrinsic) induction so as to achieve an approximately constant rate of change of B_i or B. Flexible sweep control requires a power supply for the electromagnet that can be programmed by an analog or digital electronic signal. For greatest flexibility, the power supply should be bipolar. Typical total recording times for a full hysteresis loop are between about 30 s and 5 min. Integrator drift errors can be kept acceptably small with reasonable operator care. The output voltages of the integrators and a Hall-effect field meter, if used, can be plotted directly with an analog *x,y* recorder, and salient property values are determined from this plot. Alternatively, the output voltages can be digitized, stored, and processed in a computer. Curves and calculated numerical values are then displayed on a monitor and printed out with a plotter or printer.

6. Calibration

6.1 The subsystems of the hysteresigraph for measuring field and flux quantities must be calibrated from time to time. Several alternative techniques are in common use. All ensure comparable degrees of reproducibility, but they yield strongly different absolute accuracy. The circuits for measuring flux (induction or intrinsic induction) and the magnetizing field are usually calibrated independently. However, checking hysteresigraphs against each other by remeasuring demagnetization curves of reference magnets may link these two necessary calibrations.

6.2 *Magnetic Flux and Induction*:

6.2.1 Electronic fluxmeters are conveniently calibrated by using one of the following four methods. An accuracy of ±0.1 % is achievable by the methods listed in 6.2.1.1-6.2.1.3. An error of ±5 % must be expected from the method given in 6.2.1.4. All these methods, however, calibrate only the electronic integrating and indicating/recording instrument. They leave out the hysteresigraph's sensing coils, which introduce errors because of their location relative to test specimen and electromagnet pole caps, and whose area-turns product can change as the coils age or are abused. The specimen geometry itself also affects the B_i calibration. Experience has shown discrepancies of 5 to 10 % between B_i measurements on different hysteresigraphs calibrated with volt-second standards. The four fluxmeter calibration methods are:

6.2.1.1 *Use of a volt-second generator*, consisting of a very stable source of a well-measured dc voltage and a precision timer. The level of this voltage and the length of time it is applied should be comparable to typical levels during a magnetic loop measurement with the hysteresigraph.

6.2.1.2 *Use of a mutual inductance standard*, by switching on and off a primary current measured with a precision ampere-meter. A known flux change is induced in the secondary winding of the standard, which serves as the V-s calibration signal in the fluxmeter circuit.

6.2.1.3 *Use of a search coil of precisely known area-turns*, that is moved into or removed from region of a time-constant homogeneous field, which has been measured with a nuclear magnetic resonance (NMR) gaussmeter. A rigidly constructed magnetic circuit comprising a highly stable permanent magnet with large iron pole pieces and a short air gap is a suitable field source for this. If it is well stabilized and shielded from

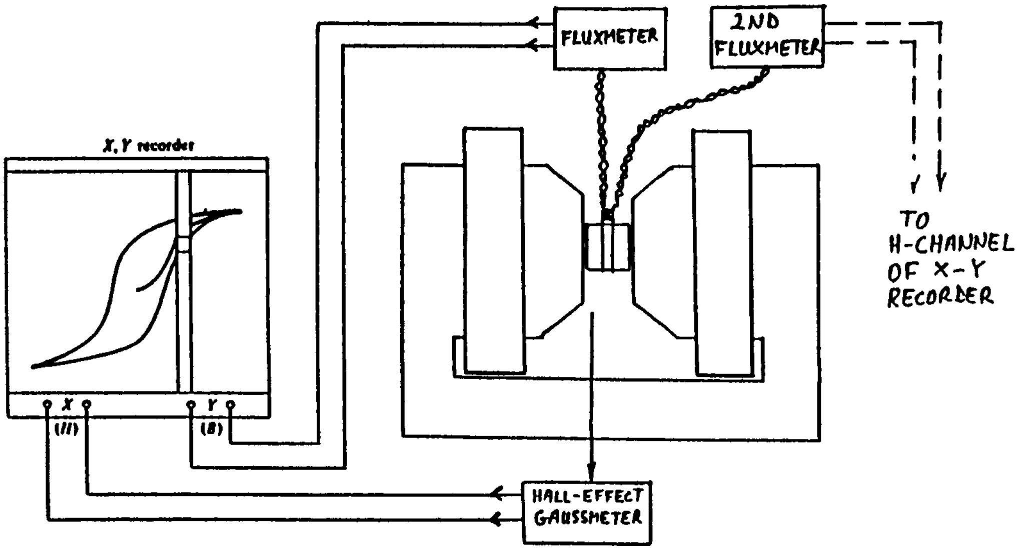

FIG. 5 Schematic Representation of a Typical Magnetic Hysteresigraph Test System

magnetic disturbances and physical abuse, it can continue to serve as a transfer standard after having once been calibrated by NMR.

6.2.1.4 *Use of the remanent induction flux*, of a long, freestanding permanent magnet bar as a secondary standard. A close-fitting, short-search coil of exactly known turns count is placed in the center (neutral zone) of the much longer bar, the fluxmeter is zeroed and the coil removed to a field-free region of space. Alternatively, the coil can be fixed and the magnet removed. The reference magnet should be precision machined from a material having a low temperature coefficient and high chemical and flux stability, such as Alnico five or temperature compensated Sm, Gd-Co-based 2-17 magnets; it must be stabilized by magnetic and thermal cycling. Its average cross-sectional area must be known.

6.2.2 The preferred method for calibrating the entire flux-measuring subsystem (B_i or B circuits, comprising the sensing coil arrangement, integrator, and indicating or recording instrument) uses a physical standard of a shape and size similar to that of the specimen to be characterized. Pure nickel is an excellent reference material since nickel is magnetically soft and thus easily saturated, its saturation magnetization value and temperature variation are well known, and nickel has a saturation induction level in the range of most permanent magnets. Pure iron is sometimes used, especially when calibrating to measure only permanent magnets with the highest induction levels. The flux calibration standard is placed in the air gap of the electromagnet, using the same pole and sensing-coil geometry to be used in the measurement for which one is calibrating. A magnetizing field of the magnitude required to produce a known magnetization in the standard is applied, and using the sensitivity potentiometers of the integrator or recorder, the y deflection on the x,y recorder is adjusted to yield a convenient scale factor for B_i. The known magnetization at the applied field value, any temperature variation of this value, and the ratio of the cross-sectional areas of standard and test specimen must be taken into account.

6.2.3 For measurements on high-B, high-H_{ci} materials, and specimens of short magnetic length, the relatively complex calibration method of 6.2.2 yields better accuracy for B_i and B than the seemingly absolute, volt-second-based fluxmeter calibration of 6.2.1. It takes into account most of the self-demagnetizing effects, field and flux inhomogeneities as a result of specimen shape and air gaps at sample end faces, and also pole-piece saturation effects, since many of these occur similarly with the nickel standard and the magnet test specimen. Experience shows the error of B_i in this case to be <2 % in the applied field range up to about 10 to 12 kOe [800 to 1000 kA/m].

NOTE 2—Pure nickel and pure iron are mechanically very soft and can be easily deformed by pressure from the electromagnet pole pieces or other forces. Such standards must be carefully protected by nonmagnetic pole spacers of matched length. They should also be frequently inspected and their dimensions carefully checked for evidence of abuse. The approach to saturation of nickel is sensitive to mechanical strain. Nickel and iron should be stress-relief annealed before being used as magnetic flux reference standards.

6.3 *Magnetic Field*:

6.3.1 The magnetic field sensor with associated instrumentation must be calibrated such that the total error in the system is within ±2 %. The method of calibration depends on the nature of the field-strength sensor used.

6.3.2 *Hall-Effect Field Meters*—These should be frequently recalibrated by placing the Hall probe in the cavity of a reference field source available from the instrument manufacturer and adjusting the electronic sensitivity controls to match the meter indication to the stated reference field strength. Such "standard magnets" comprise a stabilized permanent magnet in a small, rigidly constructed and shielded-iron circuit. They produce a stated field in the 100 to 5000 Oe [8 to 400 kA/m] range and are indirectly calibrated against a highly accurate NMR gaussmeter by their manufacturer. Hall meters can also be calibrated more directly against NMR or an accurate rotating-coil gaussmeter if a large-volume transfer magnet is available (see 6.2.1.3).

6.3.3 Some Hall probes exhibit significant nonlinearity in high fields. In this case, nominal field readings from a linear-scale meter or voltage output should be corrected using data, which the gaussmeter manufacturer normally supplies. Attention must also be paid to the often strong temperature dependence of the Hall-probe output.

6.3.4 *Inductive H-Measuring Systems Using Sensing Coils and Integrators*—The H coil may be placed in a large-volume, homogeneous and time-constant field of magnitude similar to the fields to be measured, for example, between 5 to 10 kOe [400 to 800 kA/m]. The source of this field may be a calibrated permanent magnet system (see 6.3.2) or an electromagnet with a stable current source. The field is precisely measured, the coil is then repeatedly removed and replaced while the H sensitivity of the electronic system is adjusted to match the recorder x-deflection, or other H–meter indication, to the reference field value.

6.3.5 Usually it is most convenient to produce this reference field with the hysteresigraph electromagnet and the pole-gap-coil configuration to be used in the subsequent specimen test. The field is then usually measured with a Hall gaussmeter that should be calibrated in accordance with 6.3.2. Instead of removing the coil, one can reverse the field polarity by reversing the electromagnet current.

6.4 *Simultaneous B (or B_i) and H Calibration Using Permanent Magnet Reference Specimens*:

6.4.1 Magnet producers and users often exchange permanent magnet specimens as a means of coordinating hysteresigraph measurements using magnets that are well characterized by the first party. The second party then must magnetize fully these specimens before the test and plot a demagnetization curve, repeating this procedure as needed. The sensitivity of the B or B_i measuring circuit is adjusted until the first party's B_r reading is reproduced, that of the H-measuring circuit is adjusted to reproduce the initial H_c or H_{ci} value. This is not an absolute calibration, but it is a convenient method to transfer a good calibration from one instrument to another if one party does not have the facilities for an absolute calibration.

6.4.2 The magnet material used for a secondary transfer standard must meet certain conditions. It must have sufficiently low coercivity and saturation field strength, such that each