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# Standard Test Method for Magnetic Properties of High-Coercivity Permanent Magnet Materials Using Hysteresigraphs<sup>1</sup>

This standard is issued under the fixed designation A977/A977M; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

# 1. Scope

1.1 This test method covers how to determine the magnetic characteristics of magnetically hard materials (permanent particularly their initial magnetization, magnets), demagnetization, and recoil curves, and such quantities as the residual induction, coercive field strength, knee field, energy product, 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 A341/A341M) are unsuitable. An applicable international standard is IEC Publication 60404-5.

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 operating temperature. This test method describes only how to measure 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 A34/A34M.

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 generally accepted by the 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 A2.1.

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:<sup>2</sup>
- A34/A34M Practice for Sampling and Procurement Testing of Magnetic Materials
- A340 Terminology of Symbols and Definitions Relating to Magnetic Testing
- A341/A341M Test Method for Direct Current Magnetic Properties of Materials Using D-C Permeameters and the Ballistic Test Methods
- E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

<sup>&</sup>lt;sup>1</sup> 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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<sup>&</sup>lt;sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

## 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 <sup>2</sup> ]	cm <sup>2</sup>
$\dot{B_d}$	Magnetic induction at BHmax	[T]	G
B <sub>rec</sub>	Magnetic induction at low point of recoil loop	[T]	G
Br	Magnetic induction at remanence	[T]	G
d	Diameter of pole piece	[m]	cm
d <sub>2</sub>	Diameter of homogeneous field	[m]	cm
H <sub>d</sub>	Magnetic field strength at BHmax	[A/m]	Oe
$H_p$	Magnetic field strength at low point of recoil loop	[A/m]	Oe
1	Distance between pole faces	[m]	cm
l <sub>r</sub>	Length of test sample	[m]	cm
Ň	Number of turns of test coil		
е	Voltage induced in test coil	V	V
d	Total air gap between test sample and pole faces	[m]	cm
μο	A constant with value $\mu_0 = 4\pi$ $10^{-7}$ H/m		
μ <sub>rec</sub>	Recoil permability		

2.2 Magnetic Materials Procedure Association Standard:<sup>3</sup> MMPA No. 0100–00 Standard Specifications for Permanent

Magnet Materials

 2.3 International Electrotechnical Commission Document:<sup>4</sup>
 Publication 60404-5 Magnetic Materials- Part 5: Permanent Magnet (Magnetically Hard) Materials – Methods of Measurement of Magnetic Properties

#### 3. Terminology

3.1 Basic magnetic units are defined in Terminology A340 and MMPA No. 0100–00. 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 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 5.1.5).

5.1.3 The total error of measuring *B* shall be not greater than  $\pm 2 \%$ .

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: <u>A-07(2013)</u> A B = (108(41))  $\int_{-1}^{t_2} - k((max_1, max_2, max_2$ 

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

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

where:

t

B = magnetic induction, G [T];

Δ

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;

= time, s; and

 $\int_{t_1}^{t_2} e \, dt = \text{voltage integral} = \text{flux}, \text{ V-s [Weber]}.$ 

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,  $B_{\text{corr}}$ , is given as follows:

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

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

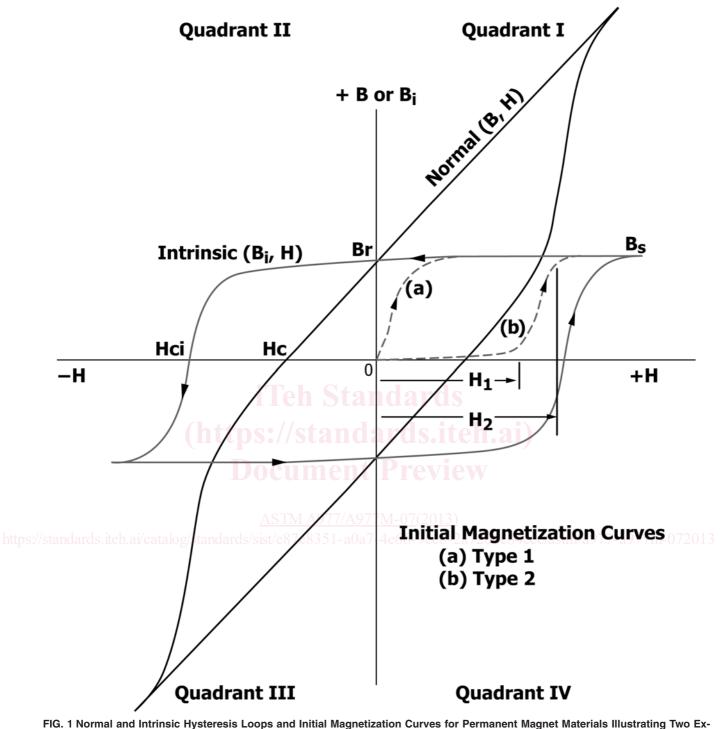
where:

- A = average cross-sectional area of the sensing coil,cm<sup>2</sup> [m<sup>2</sup>];
- $\Delta H$  = change in field from  $t_1$  until  $t_2$ , Oe [A/m]; and

<sup>&</sup>lt;sup>3</sup> Available from Magnetic Materials Producers Association, 8 S. Michigan Ave., Suite 1000, Chicago, IL 60603.

<sup>&</sup>lt;sup>4</sup> Available from International Electrotechnical Commission (IEC), 3 rue de Varembé, P.O. Box 131, CH-1211, Geneva 20, Switzerland.

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 $\mu_0$  = magnetic constant [4 $\pi$  10<sup>-7</sup> H/m].

# 5.2 Determining Intrinsic Induction :

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*. *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

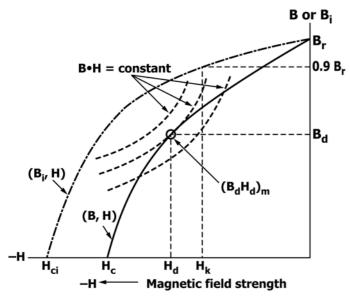


FIG. 2 Normal and Intrinsic Demagnetization Curves with Symbols for Special Points of Interest and Definition of Salient Properties. Illustration of Maximum Energy Product, Coercive Fields, and Definition of Knee Field

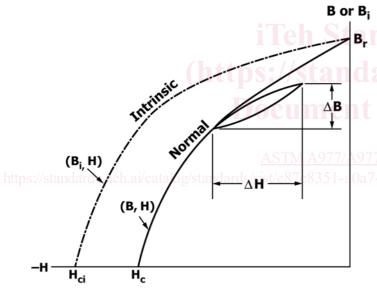


FIG. 3 Normal and Intrinsic Demagnetization Curves with Symbols for Special Points of Interest and Definition of Salient Properties. Illustration of Recoil Loop. Recoil Permeability is Defined as  $\mu_{rec} = \Delta B / \Delta H$ 

of Coil 1 except for  $B_i$  within the test specimen. The change of intrinsic induction in the specimen then is given as follows:

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

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

where:

 $B_i$  = intrinsic induction, G [T];

- $A = \text{cross section of the test specimen, cm}^2 [m^2];$  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  $\pm 2 \%$ .

# 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 Hvector 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).

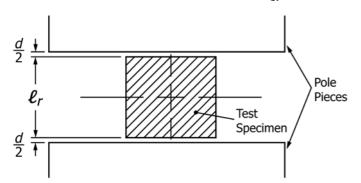


FIG. 4 Illustration Regarding the Influence of Air Gaps at the End Faces of the Test Specimen

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  $\pm 2 \%$ .

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:

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

$$\Delta H/H = 2 B d / \mu_0 l_r H (SI units)$$
(8)

To keep the error  $100 \Delta H/H < 1\%$  in the region of the  $(BH)_{\text{max}}$  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:

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, and 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  $\pm 0.1$  % is achievable by the methods listed in 6.2.1.1 – 6.2.1.3. An error of  $\pm 5$  % 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 up to 10 % between  $B_i$  measurements on different hysteresigraphs due to uncorrected sense coil and other errors, even when calibrated with volt-second standards. The four fluxmeter calibration methods are:

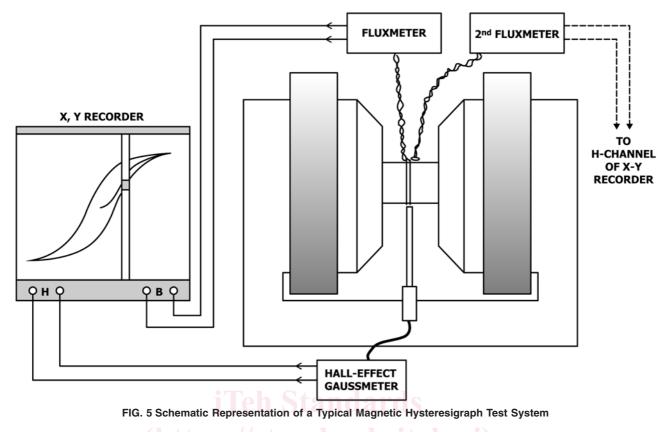
6.2.1.1 Use of a volt-second generator, consisting of a very stable source of a well-measured dc voltage and a precision timer. The level of this voltage and the length of time it is applied should be comparable to typical levels during a magnetic loop measurement with the hysteresigraph.

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 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

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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 crosssectional area must be known.

6.2.2 The preferred method for calibrating the entire fluxmeasuring 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 sensingcoil 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<sub>ci</sub>* 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  $\pm 2$  %. 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