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TECHNICAL REPORT

Methods of measurement of the magnetic properties of permanent magnet (magnetically hard) materials in an open magnetic circuit using a superconducting magnet

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METHODS OF MEASUREMENT OF THE MAGNETIC PROPERTIES OF PERMANENT MAGNET (MAGNETICALLY HARD) MATERIALS IN AN OPEN MAGNETIC CIRCUIT USING A SUPERCONDUCTING MAGNET

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68/675/DTR	68/680/RVDTR

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this Technical Report is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/standardsdev/publications.

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INTRODUCTION

Permanent magnet materials with high coercivity e.g. Nd-Fe-B magnets, have been used in industry and its usage increases rapidly to meet demands to improve energy saving and to increase efficiency of electromagnetic applications, e.g. traction motors for Electric Vehicle (EV) and Hybrid Electric Vehicle (HEV).

However, there is no standard method which can determine all the magnetic properties of the permanent magnet materials with coercivity H_{cJ} higher than 2 MA/m. The method specified in IEC 60404-5, which is a method of measurement in a closed magnetic circuit, can lead to significant measurement errors for measurement of H_{cJ} ≥ 1,6 MA/m due to magnetic saturation in parts of the pole faces of the yoke (see IEC 60404-5).

In order to solve the problem, several methods of measurement in an open magnetic circuit using a superconducting magnet (SCM) without a yoke have been developed. The methods using a SCM have been considered to be candidates for solution to accurate measurement of high performance permanent magnets.

The method using a conventional SCM made of metallic superconducting coil has not been used widely for industrial applications due to costs for using expensive liquid helium, limited speed of variation of magnetic field strength, and the difficulty to deal with test specimens of industrial size.

However, nowadays these problems have been solved thanks to the development of a ceramic SCM made of ceramic high temperature superconducting coil. This method has enabled the higher speed of variation of magnetic field strength without using precious resource of liquid helium (see Annex C). Furthermore, test apparatus using the ceramic SCM which can treat test specimens of industrial size have been commercialized globally for industrial use.

However, results of measurement in an open magnetic circuit are different from those of measurement in accordance with IEC 60404-5, particularly in terms of the squareness of demagnetization curves. This is caused by the influence of the self-demagnetizing field in the test specimen, which is opposed to magnetization. This is particular to the measurement in an open magnetic circuit. Therefore, a correction of the influence of self-demagnetizing field (demagnetizing field correction) on the demagnetization curve measured in an open magnetic circuit is indispensable.

This document describes three methods of measurement in an open magnetic circuit using a superconducting magnet (SCM), as follows:

a) SCM-Vibrating Sample Magnetometer (VSM) method;

b) SCM-Extraction method;

c) SCM-Magnetometer method.

In these methods, a test specimen is placed in a detection coil placed in a uniform magnetic field generated by a SCM. For methods a) and b), the magnetic dipole moment of the test specimen is detected by voltage induced in the detection coil due to a vibration and an extraction of the test specimen, respectively. For the method c), a variation of magnetic polarization of a stationary test specimen is detected by voltage induced in the detection coil due to a variation of the magnetic field strength applied to the test specimen.

The reproducibility of measurements of the methods a) and b) has been confirmed by an international round robin test (RRT) that was comparable with that of IEC 60404-5 (see Annex F). However, the reproducibility of the method c) has not been confirmed by a RRT yet. Therefore, the method c) is described separately in Annex A.

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There is another method of the measurement in an open magnetic circuit, i.e. the pulsed field magnetometer (PFM), which is described in IEC TR 62331 [1][1]. The PFM is different from the methods described in this document. The PFM measures a steep AC magnetic response of a test specimen in a pulsed current magnetic field. Consequently, additional correction is indispensable due to the influence of eddy currents in the test specimen and the magnetic viscosity of the magnetic materials.

A demagnetization curve should be measured by decreasing the magnetic field strength with a sufficiently slow speed during the reversal of the polarization to avoid significant magnetic viscosity and eddy current effects in accordance with IEC 60404-5. In the case of adopting a conventional metallic SCM made of metallic superconducting coil, the speed of variation of the magnetic field is too slow so that it takes an hour to obtain a demagnetization curve because of a limit of variation rate of the magnetic field to maintain the coil in a superconducting state. The problem has been solved by adopting a newly developed ceramic SCM made of ceramic high temperature superconducting coil so that a demagnetization curve can be measured within several minutes (see Annex C).

A new method of the demagnetizing field correction has been developed (see Annex E). It is a finite element method (FEM) considering the spatial distribution of self-demagnetizing field strength in the test specimen. The squareness of the corrected demagnetization curve is comparable with that measured in accordance with IEC 60404-5.

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METHODS OF MEASUREMENT OF THE MAGNETIC PROPERTIES OF PERMANENT MAGNET (MAGNETICALLY HARD) MATERIALS IN AN OPEN MAGNETIC CIRCUIT USING A SUPERCONDUCTING MAGNET

1 Scope

This Technical Report describes the general principle and technical details of the methods of measurement of the DC magnetic properties of permanent magnet materials in an open magnetic circuit using a superconducting magnet (SCM).

This method is applicable to permanent magnet materials, such as those specified in IEC 60404-8-1, the properties of which are presumed homogeneous throughout their volume.

There are two methods:

– the SCM-Vibrating Sample Magnetometer (VSM) method;
– the SCM-Extraction method.

This document also describes methods to correct the influence of the self-demagnetizing field in the test specimen on the demagnetization curve measured in an open magnetic circuit. The magnetic properties are determined from the corrected demagnetization curve.

NOTE These SCM-methods can determine the magnetic properties of permanent magnet materials with coercivity higher than 2 MA/m. The methods of measurement in a closed magnetic circuit specified in IEC 60404-5 can lead to significant measurement error due to saturation effects in the pole pieces of yoke for the magnetic materials with coercivity higher than 1,6 MA/m (see IEC 60404-5).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60404-5, *Magnetic materials – Part 5: Permanent magnet (magnetically hard) materials – Methods of measurement of magnetic properties*

IEC 60404-8-1, *Magnetic materials – Part 8-1: Specifications for individual materials – Magnetically hard materials*

IEC 60050-121, *International Electrotechnical Vocabulary – Part 121: Electromagnetism*

IEC 60050-151, *International Electrotechnical Vocabulary – Part 151: Electrical and magnetic devices*

IEC 60050-221, *International Electrotechnical Vocabulary – Chapter 221: Magnetic materials and components*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in IEC 60050-121, IEC 60050-151, IEC 60050-221 and the following apply.

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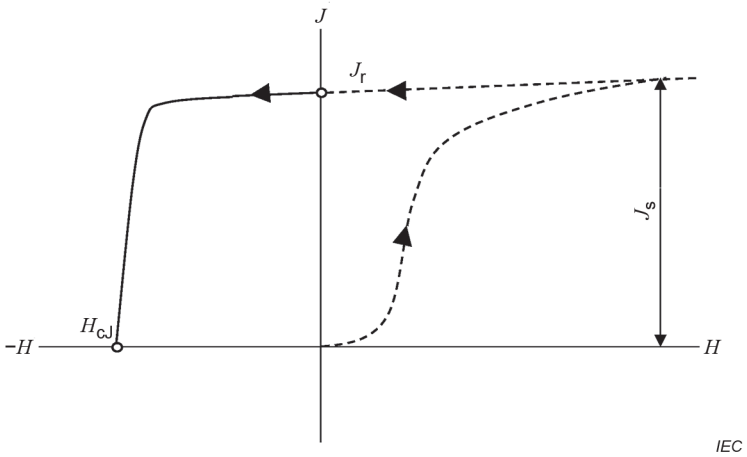
- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1
demagnetization curve
part of a hysteresis loop in which the magnetic polarization goes from the remanent magnetic polarization to zero when the applied magnetic field strength varies monotonically, as illustrated in Figure 1



Key

J_s saturation magnetic polarization, in T

J_r remanent magnetic polarization, in T

H_{cJ} coercivity relating to the magnetic polarization, in A/m

Figure 1 – Demagnetization curve $J(H)$

Note 1 to entry: A demagnetization curve can be measured from near magnetic saturation.

[SOURCE: IEC 60050-121:1998, 121-12-72, modified – magnetic flux density is replaced by magnetic polarization and Note 1 to entry and Figure 1 have been added]

3.2
magnetic dipole moment
m
vector quantity given by the volume integral of the magnetic polarization

[SOURCE: IEC 60050-221:1990, 221-01-07, modified – the symbol j is changed to m which is used industrially and the note has been removed]

3.3
***M* coil**
detection coil for magnetic dipole moment

3.4
***J* coil**
detection coil for magnetic polarization

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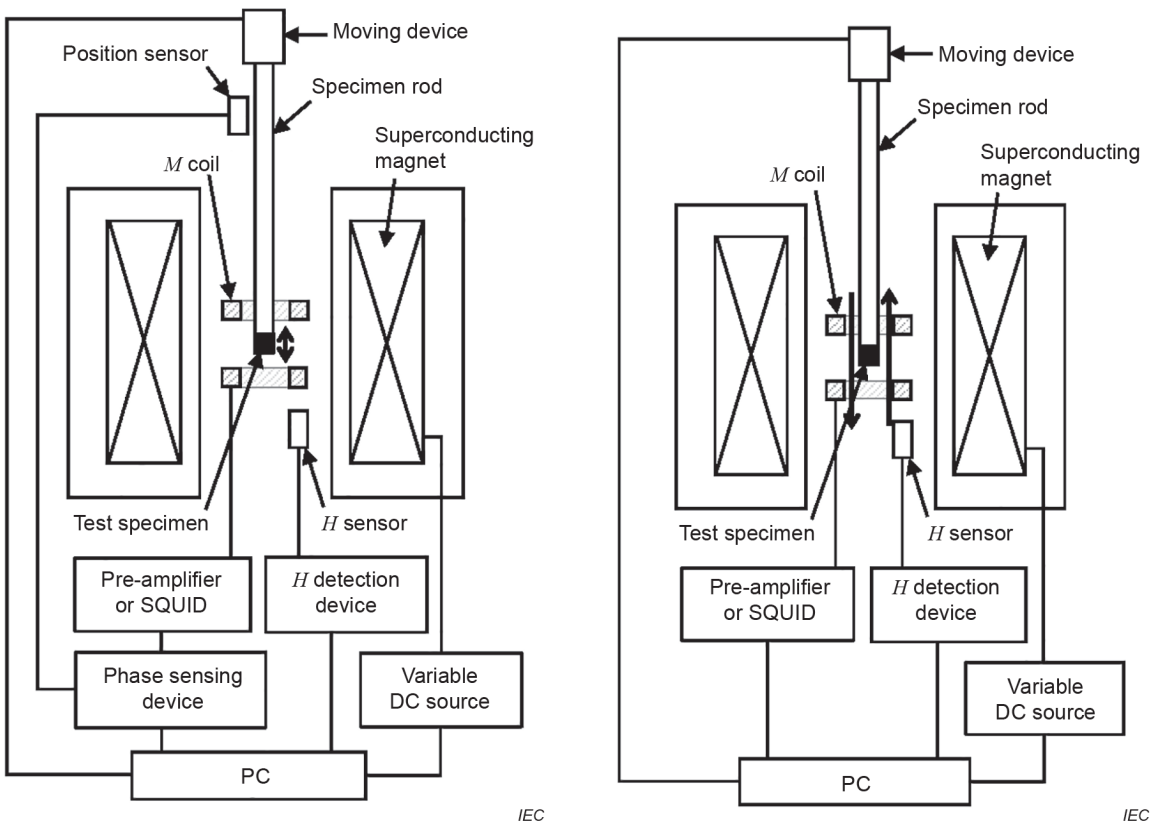
4 General principle

4.1 Principle of the method

Figure 2 illustrates schematic diagrams of typical test apparatuses. The test apparatus consists of a superconducting magnet (SCM), a moving device, a specimen rod, a magnetic field sensor (hereafter H sensor), a magnetic dipole moment detection coil (hereafter M coil), measuring devices and a data processing device (PC). The measurement is carried out in an open magnetic circuit to enable the determination of magnetic properties of permanent magnet materials with coercivity higher than 2 MA/m.

The axis of the DC magnetic field generated by the SCM is vertical and coaxial with the M coil and the specimen rod. The moving test specimen is placed in a zone where the magnetic field strength is uniform with a tolerance of ±1 % at the centre of the SCM. The H sensor is placed in a zone where the influence of the magnetic dipole moment of the test specimen can be ignored.

A test specimen is firmly attached on the specimen rod so that the direction of magnetization is parallel to the axis of the specimen rod, and then placed in the test apparatus as shown in Figure 2.



a) The SCM-VSM method

b) The typical SCM-Extraction method

Figure 2 – Schematic diagrams of the test apparatus

The test specimen is initially magnetized to saturation (see 6.2), and then a DC magnetic field is applied to the test specimen in the direction opposite to that used for the initial magnetization. The magnetic field strength is measured by the H sensor (see 4.3).

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The magnetic dipole moment of the test specimen is detected by the voltage induced in the M coil due to the movement of the test specimen (see 4.4). The magnetic polarization of the test specimen is calculated from the magnetic dipole moment and the volume of the test specimen (see 7.2). For calibration aspects, see Clause 9.

There are two methods different in modes of the movement of the test specimen:

a) the SCM-VSM method: the test specimen is vibrated with a small amplitude in the M coil;

b) the SCM-Extraction method: the test specimen is extracted through the M coil.

NOTE There is another method to determine the magnetic polarization of the test specimen, i.e. the SCM-Magnetometer method. In this method, variation of the magnetic polarization of the stationary test specimen due to variation of the magnetic field strength applied to the stationary test specimen is detected by the voltage induced in the detection coil (J coil) (see Annex A).

The measurements are carried out at an ambient temperature of (23 ± 5) °C. For permanent magnet materials which are known to have significant temperature coefficients $\alpha(J_r)$ and $\alpha(H_{cJ})$, the temperature of the test specimen should be in a range between 19 °C and 27 °C and controlled within a tolerance of ±1 °C during the measurements in accordance with IEC 60404-5. The temperature of the test specimen should be measured by a non-magnetic temperature sensor.

The demagnetization curve measured in an open magnetic circuit is influenced strongly by the self-demagnetizing field in the test specimen which opposes magnetization.

In order to determine the intrinsic demagnetization curve of the permanent magnet material, a correction of the influence of the self-demagnetizing field (hereafter demagnetizing field correction) should be applied to the measured demagnetization curve (see Clause 11). Magnetic properties of the permanent magnet material are determined from the corrected demagnetization curve.

These two methods have the following features:

1) The most important feature is that it is possible to determine all the magnetic properties of permanent magnet materials with coercivity higher than 2 MA/m in contrast to the method of measurement in a closed magnetic circuit in accordance with IEC 60404-5.

2) The reproducibility of measurement by the methods is comparable with that of IEC 60404-5. It was confirmed by the international round robin test (see Annex F).

3) The influence of eddy currents in the test specimen is negligible.

4) By adopting the ceramic SCM made of ceramic high temperature superconducting coil, demagnetization curve can be measured within several minutes without using expensive liquid helium and its incidental facilities (see Annex C). Also test apparatus using the ceramic SCM which can deal test specimens of industrial size has been commercialized for industrial use. So, it is convenient for industrial use globally.

5) There is no drift in the signal of the magnetic dipole moment, owing to the use of a phase sensing device (lock-in amplifier) in the SCM-VSM.

4.2 Superconducting magnet (SCM)

A variable DC source supplies a DC current to the superconducting coil, with sufficiently low voltage noise (see Figure 2). The current source should be a bipolar type which can switch positive-negative polarity continuously.

The SCM is recommended to have a capacity to generate a magnetic field strength more than 4,8 MA/m (6 T in magnetic flux density) in order to measure the magnetic properties of permanent magnet materials with coercivity higher than 2 MA/m, e.g. Nd-Fe-B sintered magnets.

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It is recommended to adopt the ceramic SCM made of ceramic high temperature superconducting coil rather than a conventional metallic SCM made of metallic superconducting coil, in order to reduce the time required to measure a demagnetization curve within several minutes. It is particularly convenient for industrial use (see Annex C).

The zone of uniform magnetic field strength generated at the centre of the SCM should be sufficiently large to include the space of the moving test specimen.

4.3 Magnetic field strength sensor (*H* sensor)

An *H* sensor such as a Hall probe is used to measure the magnetic field strength together with a suitable *H* detection device (see Figure 2). The *H* sensor should be calibrated by an appropriate method such as Nuclear Magnetic Resonance (NMR).

In the case of a calibrated SCM, the magnetic field strength may be measured from the magnetizing current supplied to the SCM. Care should be taken if there is a small hysteresis between the magnetizing current and the magnetic field strength of the SCM.

The total measuring error of the magnetic field strength should be smaller than ±1 %.

4.4 Magnetic dipole moment detection coil (*M* coil)

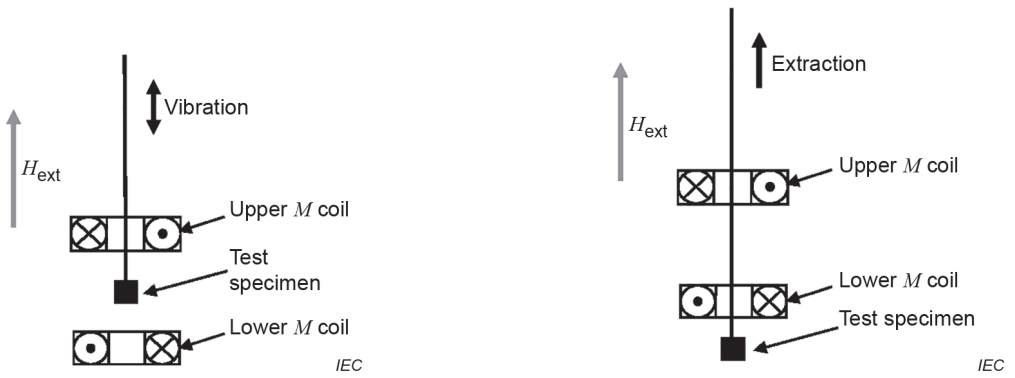
The magnetic dipole moment of the test specimen is measured by the voltage induced in the *M* coil placed near the test specimen (see Figure 2). The *M* coil is wound coaxially with the axis of magnetic field and placed symmetrically with respect to the centre of the magnetic field. Electrical leads of the *M* coil should be tightly twisted to avoid errors caused by voltages induced in loops of the leads.

The voltage induced in the *M* coil should be calibrated using a standard specimen of nickel sphere and the influence of the shape and dimensions of the test specimen on the voltage should be verified (see Clause 9).

The total measuring error of the magnetic dipole moment should be smaller than ±1 %.

The *M* coil used in this document is the first order gradiometer coil which is composed of an upper coil and a lower coil connected electrically in opposite polarity as shown in Figure 3. The second order gradiometer coil combined with a SQUID (superconducting quantum interference device) circuit can also be used for the *M* coil (see Annex D).

NOTE Figure D.2 illustrates the dependence of the induced voltage on the position of the test specimen in the SCM-Extraction method.



a) The SCM-VSM method

b) The typical SCM-Extraction method

Figure 3 – Schematic diagrams of the first order gradiometer coil

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