

Superconductivity —

Part 9: Measurements for bulk high temperature superconductors — Trapped flux density of large grain oxide superconductors

The European Standard EN 61788-9:2005 has the status of a British Standard

ICS 17.220; 29.050

National foreword

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The UK participation in its preparation was entrusted to Technical Committee L/-/90, Superconductivity, which has the responsibility to:

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Superconductivity
Part 9: Measurements for bulk high temperature superconductors -
Trapped flux density of large grain oxide superconductors
(IEC 61788-9:2005)

Supraconductivité
Partie 9: Mesures pour supraconducteurs
haute température massifs –
Densité de flux résiduel des oxydes
supraconducteurs à gros grains
(CEI 61788-9:2005)

Supraleitfähigkeit
Teil 9: Messungen an massiven
Hochtemperatursupraleitern -
Eingefrorene magnetische Flussdichte
bei grobkörnigen oxidischen Supraleitern
(IEC 61788-9:2005)

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Comité Européen de Normalisation Electrotechnique
Europäisches Komitee für Elektrotechnische Normung

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Foreword

The text of document 90/167/FDIS, future edition 1 of IEC 61788-9, prepared by IEC TC 90, Superconductivity, was submitted to the IEC-CENELEC parallel vote and was approved by CENELEC as EN 61788-9 on 2005-06-01.

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Endorsement notice

The text of the International Standard IEC 61788-9:2005 was approved by CENELEC as a European Standard without any modification.

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INTRODUCTION

Large grain bulk high temperature superconductors (BHTSC) have significant potential for a variety of engineering applications, such as magnetic bearings, flywheel energy storage systems, load transports, levitation, and trapped flux density magnets. Large grain superconductors have already been brought to market worldwide.

For industrial applications of bulk superconductors, there are two important material properties. One is the magnetic levitation force, which determines the tolerable weight supported by a bulk superconductor. The other is the trapped flux density, which determines the maximum field that a bulk superconductor can generate. The users of bulk superconductors must know these values for the design of their devices. However, these values are strongly dependent on the testing method, and therefore it is critically important to set up an international standard for the determination of these values both for manufacturers and industrial users.

The test method covered in this standard is based on the VAMAS (Versailles Project on Advanced Materials and Standards) pre-standardization work on the properties of bulk high temperature superconductors.

SUPERCONDUCTIVITY –

Part 9: Measurements for bulk high temperature superconductors – Trapped flux density of large grain oxide superconductors

1 Scope

This part of IEC 61788 specifies a test method for the determination of the trapped field (trapped flux density) of bulk high temperature superconductors.

This International Standard is applicable to large grain bulk oxide superconductors that have well defined shapes such as round discs, rectangular, and hexagonal pellets. The trapped flux density can be assessed at temperatures from 4,2 K to 90 K. For the purpose of standardization, the trapped flux density will be reported for liquid nitrogen temperature.

2 Normative references

The following referenced documents are indispensable for the application 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 60050(815):2000, *International Electrotechnical Vocabulary – Part 815: Superconductivity*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in IEC 60050(815) and the following apply.

3.1

trapped flux density

strength of the magnetic flux density (T) trapped by a bulk high temperature superconductor (BHTSC) at a defined gap and at a defined temperature

3.2

maximum trapped flux density

peak value of the trapped flux density

NOTE For most measurements, only the z component of the flux density is measured, which is strongly affected by the sample geometry or the demagnetizing effect (see Clause A.2). Thus the total flux density, which is the integration of all the field components, may also be regarded as the materials property to stand for the trapped flux density (see Clause A.1).

4 Principle

Superconductors that exhibit flux pinning are capable of trapping magnetic fields, as shown in Figure 1. Here the internal magnetic flux density rotation ($\nabla \times \mathbf{B}$) in the BHTSC is proportional to the critical current density (J_c), as expressed by the following equation:

$$\nabla \times \mathbf{B} = \mu_0 \mathbf{J}_c$$

In one dimension, the equation is reduced to

$$dB_z/dx = \mu_0 J_c^y$$

in rectangular coordinates or to

$$dB_z/dr = \mu_0 J_c^\theta$$

in cylindrical coordinates.

The maximum value of the trapped flux density in the z component ($B_{z,max}$) in an infinite cylinder (2 R in diameter) is given by the following equation:

$$B_{z,max} = \mu_0 J_c^\theta R$$

In practical samples, this value is reduced by the demagnetizing effect or the geometrical effect as follows:

$$B_{z,max} = D(R/t) \mu_0 J_c^\theta R$$

where $D(R/t)$ is a geometrical constant that depends on the shape (the ratio of radius/thickness) of the BHTSC.

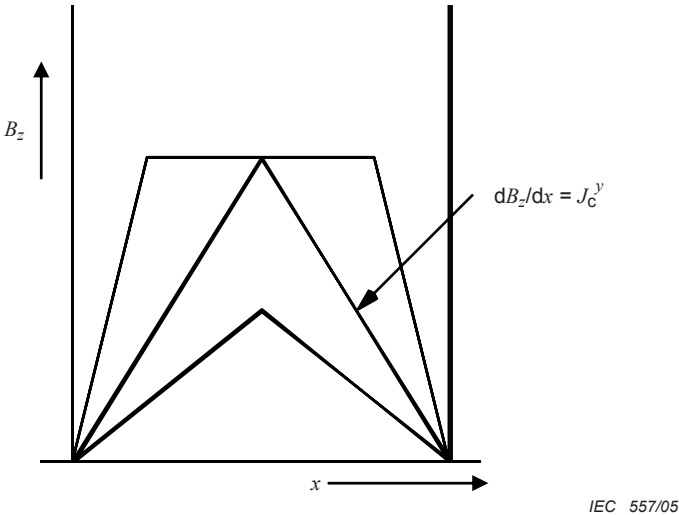
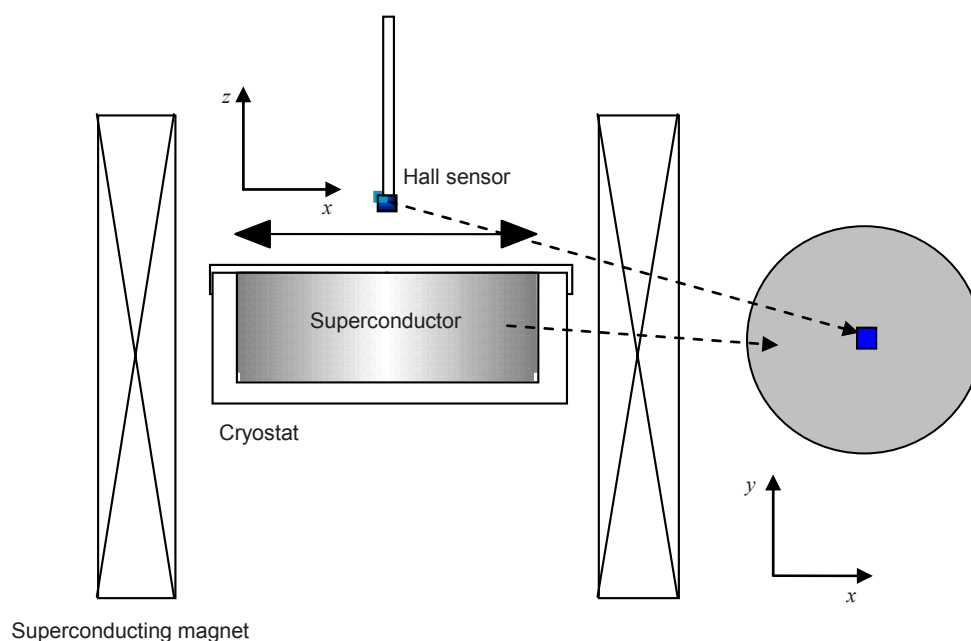


Figure 1 – Principle of trapped flux density in bulk superconductor

Figure 2 shows a schematic diagram of the experimental set-up for trapped flux density measurements [1]¹⁾. There are several ways to measure the trapped flux density of BHTSC. A typical measurement procedure is as follows. Firstly, the field is applied on the superconductor. Secondly, the sample is fixed on the cold head of a cryostat, which is cooled to the target temperature by using a cooling device. After reaching the target temperature, the external field is removed. The distribution of the field trapped by the BHTSC is then measured by scanning a Hall sensor over the specimen surface at a defined gap. This is the so-called field-cooled (FC) method of magnetization.



IEC 558/05

Figure 2 – Schematic view of the experimental set-up

5 Requirements

Upon removal of the external field, the trapped flux density will decay with time from its initial value. This is due initially to flux flow and later to flux creep (collectively termed flux relaxation). The initial peak value shall not be used for the design of machines.

The trapped flux density values are those measured after a sufficiently long time has passed since the appropriate measurement conditions were reached. The trapped flux density values shall be measured at least 15 min after the external field is removed from the specimen under test.

The target precision of this method is that the coefficient of variation in any inter-comparison test shall be 5 % or less for measurements performed within 1 month of each other [2].

It is the responsibility of the user of this standard to consult and establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given below.

1) Figures in square brackets refer to the bibliography.

Hazards exist in this type of measurement. Very large direct currents with very low voltages do not necessarily provide a direct personal hazard, but strong magnetic fields trapped by the BHTSC may cause the problem. It is imperative to shield magnetic fields. Also the energy stored in the superconducting magnets commonly used for generating the magnetic field can cause large current and/or voltage pulses, or deposit a large amount of thermal energy in the cryogenic systems causing rapid boil-off or even explosive conditions. Direct contact of skin with cold liquid transfer lines, storage dewars or apparatus components can cause immediate freezing, as can direct contact with a spilled cryogen. It is imperative that safety precautions for handling cryogenic liquids be observed.

6 Apparatus

6.1 Cryostat

The cryostat shall include a BHTSC specimen support and a liquefied cryogen reservoir for the measurements. Other cooling devices can also be used for the temperature control of the specimens. Before measurements, the specimen shall be held at the measured temperature for a sufficient amount of time to cool, since large grain BHTSC specimens in typical size (greater than 3 cm in diameter) require a long time for the entire body to reach the target temperature. The recommended waiting time can be estimated by considering the size and thermal conductivity coefficient of the BHTSC. For a large grain BHTSC, the temperature tends to increase during the measurements, so the power of the cooling device shall be large enough to avoid a temperature rise of the specimen.

6.2 Activation magnet

In principle, any activation magnet or a magnetizing device can be used as long as the trapped flux density is saturated (see Clause A.3).

The activation magnet shall have a working area larger than the dimension of BHTSC. The magnetizing field required to saturate the field trapping ability of BHTSC is determined by the demagnetizing factor of the sample (see Clause A.3). If the field strength of the activation magnet is high enough, the applied field does not need to be uniform.

Pulse field activation is not recommended for standardization, since the error associated with this magnetization process is very large and its results are generally non-reproducible.

6.3 Support of BHTSC

During trapped flux density measurements, large electromagnetic forces will act on the BHTSC. Therefore, the BHTSC shall be firmly fixed to the support, which shall be non-magnetic and have a high enough mechanical strength to withstand the electromagnetic force. The BHTSC shall be fixed to the support, in most cases, with materials that harden at low temperatures. If the uniformity of the BHTSC is sufficiently good with the c -axis aligned to the external field, the measurements can be performed by simply placing the BHTSC on a non-magnetic substrate.

Due to the large anisotropy, induced currents mainly flow within the a - b plane. When the c -axis is not parallel to the external field, a large torque acts on the BHTSC so as to align the c -axis of the specimen parallel to the direction of external field. The BHTSC often tilts with such torque force that an extra support is necessary to withstand the torque.

A large electromagnetic force acts on the BHTSC during the measurements, which sometimes leads to fracture. BHTSC is a ceramic material and intrinsically brittle, furthermore it contains a large amount of pores and cracks, which deteriorates the mechanical properties of BHTSC. Thus the measurement might lead to the destruction of the BHTSC. The manufacturer can improve the mechanical properties by reinforcement (see Clause A.4).

6.4 Field mapping unit

A field mapping unit consisting of a magnetic Hall sensor or arrangements of magnetic Hall sensors mounted on a two-axis translational device shall be used. The sensing area of the Hall sensor shall be <2 % of the area of the specimen and shall have sensitivity <0,001 T. The translation range of the device shall be larger than the largest dimension of the specimen in the x - y scanned plane.

The measured trapped field strength is dependent on the distance between the top surface of the superconducting specimen and the Hall sensor element. The distance, which includes the thickness of the encapsulating resin and/or layer of reinforcement, shall be kept at <10 % of the specimen thickness.

6.5 Temperature measurements

The temperature of the BHTSC shall be measured with a suitable temperature sensor. The sensor shall be mounted on the support plate as closely to the sample as possible. Temperature sensors that are influenced by magnetic fields shall be avoided.

7 Measurement procedure

The BHTSC shall be cooled in the presence of a static magnetic field generated by the magnet discussed in 6.2 (field-cooled). When the specimen has been completely cooled, the activation field shall be removed or reduced to zero. In order to avoid a strong influence of flux flow and flux creep on the measurements, the specimen shall be allowed to settle for at least 15 min before measurements are performed.

The distribution of magnetic field trapped by BHTSC shall be measured with a magnetic Hall sensor. The sensor shall be scanned over the x - y plane of the specimen measuring the z component of magnetic field over a predetermined grid while maintaining a certain gap between the sensor element and the specimen surface. The grid spacing shall be <10 % of the largest dimension of the x - y plane that is being scanned. If the field distribution is symmetric across every diameter within 10 %, the peak value shall be regarded as the trapped flux density.

Alternatively, arrangements of magnetic Hall sensors can be used to measure the trapped flux density of the specimen. If the spacing of the sensors is small enough, and the entire specimen is covered by the sensors, scanning is not necessary.

Careful calibration of the magnetic Hall sensor shall be performed at operating temperature. The temperature near the Hall sensor shall be monitored and used to correct the data with the Hall sensor calibration curve.

8 Precision and accuracy of the test method

8.1 Temperature

The liquid nitrogen temperature shall be determined to an accuracy of $\pm 0,25$ K, while holding the specimen, which is mounted on the measuring base plate.

8.2 Field

The external magnetic field shall be determined to an accuracy of $\pm 0,05$ T. The magnetic sensor used for the field mapping shall be accurate within $\pm 0,05$ T.

8.3 Gap distance

The distance between the top surface of the superconducting specimen and the bottom of the Hall sensor element, which includes the thickness of the encapsulating resin, shall be determined to an accuracy of ± 10 %.

9 Test report

The following items shall be reported if known.

9.1 Specimen

The test specimen shall be identified, if possible, by the following information.

- a) Shape and dimensions
- b) Post growth treatment (reinforcement, irradiation etc.)

9.2 Test conditions

The following test conditions shall be reported.

- a) Activation magnet
The maximum field, the bore diameter (or sample diameter for BHTSC magnet)
- b) Time to reduce the external field to zero
- c) Waiting time to start measurements after the removal of the external field
- d) Specification of magnetic field sensor
- e) Kind, size, activation area, calibration curves, sensitivity
- f) Locations of field sensor
- g) Installation method of the specimen on the base plate
- h) Materials, shape and dimensions of the base plate
- i) Specification of cryostat
- j) Type(s) of thermometers
- k) Locations of thermometers with respect to the BHTSC

9.3 Trapped flux density

The following information should be provided.

- a) Trapped flux density
- b) Gap (between the bottom of the Hall sensor and the top of the sample surface)
- c) Temperature
- d) Applied activation field
- e) Field distribution map (optional)

Annex A (informative)

Additional information related to Clauses 3 to 6

A.1 Definition of term

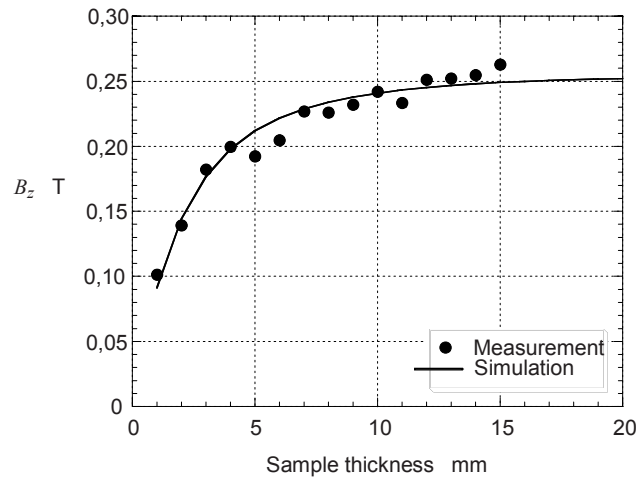
Total trapped flux density

For industrial standard, we measure the distribution of the z component of trapped flux density. However, due to the demagnetizing effect, the z component is strongly affected by the geometry or the aspect ratio of a BHTSC. If one can measure all the components of trapped flux density, which are B_x , B_y , B_z , and the demagnetizing effect can be neglected,

$$|B| = \sqrt{B_x^2 + B_y^2 + B_z^2}$$
 is termed the total flux density.

A.2 Geometrical effect on trapped flux density

The trapped flux density is strongly dependent on the sample geometry, especially the aspect ratio or the diameter/thickness ratio (see Figure A.1). Under constant J_c - B properties, the trapped flux density first increases with increasing thickness and saturates at a certain value. Thus an inter-comparison of different samples should be performed with the same dimensions, otherwise a correction of the geometry effect is necessary.



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Figure A.1 – Thickness dependence of the trapped flux density (B_z)

NOTE 1 Samples are 15 mm in diameter [3].

NOTE 2 The trapped flux density increases with increasing thickness and then saturates.

A.3 Activation magnet

Normal conducting electromagnets can be used for activation, however, it should be confirmed that the maximum trapped flux density of the BHTSC is smaller than the activation field.

It is important for the activation magnet to have a working area larger than the dimension of BHTSC, in which a constant field is applied to BHTSC either by an electromagnet or by another BHTSC. The field generated by the magnet needs to be high enough to saturate the field trapping ability of BHTSC. If the field strength of the activation magnet is high enough, the applied field does not need to be uniform. The maximum field should be determined by considering the saturation field and demagnetizing effects associated with the sample dimensions. For example, an applied field of at least 1,75 times the maximum trapped field is required to fully magnetize a superconducting sample with a width to thickness aspect ratio of 2,5 [2]. This level of magnetizing field increases with increasing the ratio.

A.4 Reinforcement of BHTSC

During trapped flux density measurements, the BHTSC specimen experiences a large thermal stress and a large electromagnetic pressure. Since the BHTSC is a brittle ceramic, it often fractures due to the stresses. Hence it is desirable to reinforce the BHTSC not only for standardization but also for industrial applications.

Reinforcements with metal rings are commonly employed. Recently, resin impregnation has been found to be effective in improving the mechanical properties of BHTSC. In this method, the BHTSC is immersed in molten resin and placed in a vacuum. The resin fills open cracks and voids near the surface, leading to a dramatic improvement of mechanical properties.

A.5 Magnetic sensor

Any magnetic sensor can be used for the measurements of trapped flux density. Hall sensors and pick-up coils have been commonly used for the measurements. Hall sensors often have temperature dependence, so the temperature of the Hall sensor needs to be known along with the calibration method used. One way to ensure the temperature of the Hall sensor is to operate it in liquid nitrogen.

A.6 Extrapolation to zero gap

For fair comparison of the trapped flux density, one may extrapolate the trapped flux density value B_z at a finite gap (z) to zero gap using the following equation in the case of a cylinder:

$$B_z(z) = C \left((z+D) \ln \frac{R + \sqrt{R^2 + (z+D)^2}}{z+D} - z \ln \frac{R + \sqrt{R^2 + z^2}}{z} \right)$$

where C is the constant related to the critical current density, R is the radius and D the height of the cylinder [4]. For example, in the cylindrical sample 30 mm in diameter and 15 mm in thickness with the peak value of the trapped flux density of 2,6 T at zero gap, the trapped flux density decays with gap as shown in Figure A.2. But one should notice that this method is a first order approximation, and will not be a good approximation when the field dependence of the critical current density is significant.

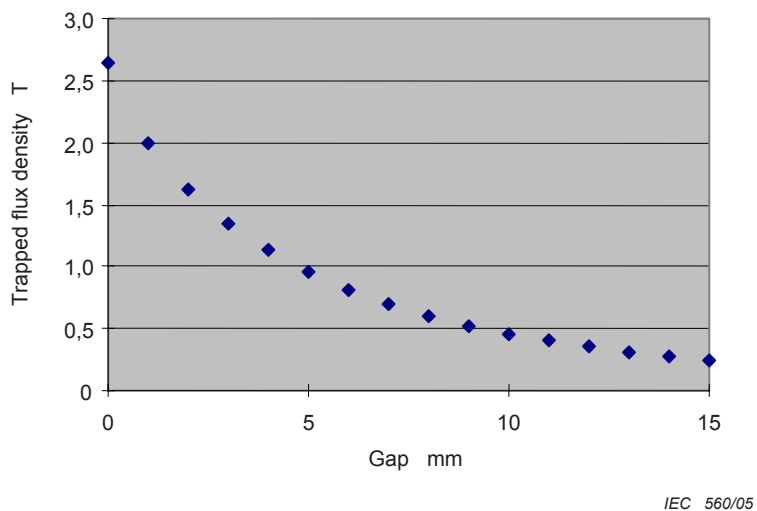


Figure A.2 – Gap dependence of the field strength

NOTE 1 The sample is 30 mm in diameter and 15 mm in thickness. The maximum trapped flux density is 2,6 T at zero gap.

NOTE 2 One may be able to determine the trapped flux density value B_z at any arbitrary gap (z) using the data at another gap.

Annex B (informative)

Measurements for levitation force of bulk high temperature superconductors

B.1 Principle

The levitation force is often used for characterizing the BHTSC. The force measurements are much easier than the trapped flux density measurements, and thus more widely used. However, the levitation force is essentially limited by the field strength and the spatial distribution of a permanent magnet, which is commonly used for the force measurements.

B.2 Apparatus

B.2.1 Permanent magnet

A permanent magnet (PM), which has identical magnetic properties (BH_{\max} , B_r , and B_c) and identical dimensions (radius and height) should be used for a standard test method. When the size of the BHTSC is not the same, at least, the dimensional ratio of PM/BHTSC should be maintained constant for comparison.

Special care should also be paid to maintaining a temperature of the PM constant during the force measurements, since magnet properties of the PM are strongly temperature dependent. For this purpose, the PM should be covered with epoxy resin or other materials with small thermal conductivity. It is also desirable to thermally insulate the cryostat, in which the BHTSC is installed, or to thermally shield the PM.

B.2.2 The support of the bulk superconductor

During force measurements, the BHTSC should be firmly fixed to the support, since a large electromagnetic force will act on the superconductor. The support and the BHTSC are cooled in a cryostat, whose temperature should be kept constant during measurements. As mentioned above the cooling power of the cryostat should be large enough to avoid the temperature rise of the BHTSC. The mechanical strength of the support should be large enough to avoid the motion of the BHTSC during force measurements. In most cases, the BHTSC is glued to the support with a material that hardens with decreasing temperature. It is desirable to further fix the BHTSC to the support using a non-magnetic metal sheath.

B.2.3 Driving unit of the PM

The levitation forces are measured as the PM is moved toward and away from the BHTSC. The approach speed of the PM should be low, since it strongly affects the force values, and also the decay due to flux flow and flux creep.

B.2.4 Force measurement unit

The levitation force should be measured using a standardized strain gauge. The tensile testing machine type force measuring system is recommended.

B.3 Test report

The levitation force should be measured as a function of the gap between the PM and the BHTSC. The initial gap should be large enough to avoid the field of the PM being trapped by the BHTSC. First the force is measured as the PM approaches to the BHTSC, and then the force is measured as the PM moves away from the BHTSC. After the cycle, the BHTSC should be warmed above the critical temperature, since magnetic flux is always trapped by the BHTSC during the force measurements. This trapped flux would affect the force values in the second cycle. The force and the gap should be recorded with a computer at an identical approaching speed of the PM toward and away from the BHTSC.

Force measurements should be repeated by gradually reducing the approach speed of the PM each time. If the approach is slow enough, the force versus gap curves become the same. Here, the levitation force may be defined as the value at a defined gap. However, the force value, which can be used for machine design, will be smaller than this value. For obtaining such a value, the PM approaches to the defined gap at enough speed and is left there for 1 h while monitoring the decay of the levitation force. The levitation force is then defined as the value held at the gap for at least 1 h.

In order to make results more general it is also possible to report the force as a fraction of the theoretical maximum, that is the force obtained when the BHTSC behaves as a perfect diamagnet. In this case, the force measurement conditions are not so critically important. The problem is how to calculate the theoretical maximum. (PM companies have developed their own computer programs to obtain this value.)

B.4 Relationship between the trapped magnetic field and the levitation force

Trapped flux density values have a strong correlation to the levitation force only when the field strength used for force measurements is large enough for the external field to reach the centre of the specimen. Such a condition is not fulfilled in most experiments, where the levitation force of large grain BHTSC is measured using a conventional permanent magnet. However, the trapped flux density values may be converted to the levitation force values, once the field dependence of macroscopic J_c is known.

The magnetization of a superconductor is given by the following relation:

$$M(H) = AJ_c(B)d$$

where M is the magnetization, A is a geometrical constant, J_c the critical current density and d is the characteristic length scale of the supercurrent loop. This equation applies to both the levitation force and the trapped flux density measurements under the condition that supercurrents are flowing in the entire body. Such a condition is easily fulfilled in the trapped flux density measurements, however not for the force measurements, in which the external field does not reach the centre of the BHTSC. In the state of partial penetration, M is given by

$$M = -H + \frac{H^2}{J_c d}$$

where H is the external field. Therefore, M is not a simple function of materials parameters of J_c and d . Furthermore, the levitation force also depends on the field gradient as

$$F_z = M_z \frac{dH_z}{dz}$$

where the subscript z stands for rectangular coordinate component of these variables.

However, one can obtain the field dependence of J_c from the results of trapped flux density measurements, which can be used to calculate the levitation force.

Annex C (informative)

Test report (example)

C.1 Specimen

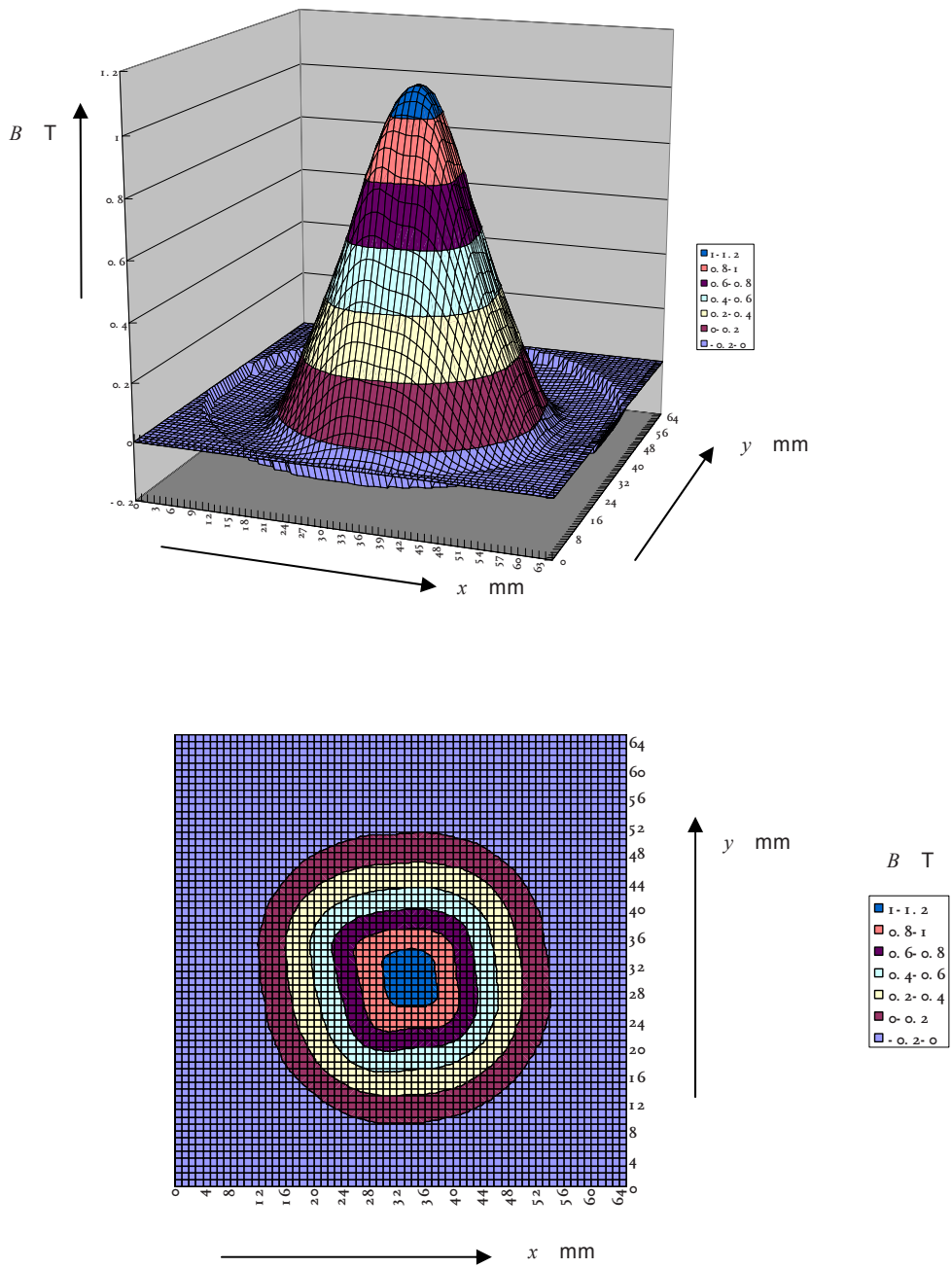
- a) Shape and dimensions: 46 mm in diameter, 15 mm in height.
- b) Post growth treatment: epoxy resin impregnation (0,5 mm in thickness).

C.2 Test conditions

- a) Activation magnet: 10 T superconducting magnet (NbTi, Nb₃Sn hybrid type), 10 cm room temperature bore.
- b) Time to reduce the external field to zero: 10 min (2 T to zero).
- c) Waiting time: 20 min.
- d) Specification of a magnetic sensor: low temperature Hall sensor (FW Bell, BHA-921, sensitivity 0,8 mV/kG, axial type, 6,35 mm diameter, 5,08 mm in thickness, active area 0,5 mm in diameter).
- e) Locations of field sensor: the Hall sensor was scanned an area of 50 mm × 50 mm with at step size of $\Delta x = \Delta y = 0,5$ mm.
- f) Installation method of the specimen on the base plate: glued to the FRP plate of a cryostat with silicone grease.
- g) Materials, shape and dimensions of the base plate: copper plate 60 mm in diameter 5 mm in thickness.
- h) Specification of cryostat: made of FRP, 99 mm in outer diameter, 90 mm in inner diameter, 210 mm in height. The base plate was FRP 10 mm in thickness. The cryostat was filled with liquid nitrogen.
- i) Type(s) of thermometers: GaAIAs Diode (Lakeshore TG-120).
- j) Locations of thermometers with respect to the BHTSC: the side of the sample. Inside liquid nitrogen in the cryostat.

C.3 Trapped flux density

- a) Trapped flux density: 1,1 T at the peak.
- b) Gap: 1,0 mm (including the mould thickness of 0,3 mm). The value at zero gap can be estimated to be according to the equation given in Clause A. 6.
- c) Temperature: 77,5 K.
- d) Applied activation field: 2 T.
- e) Field distribution map.



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Figure C.1 – Distribution map of trapped flux density

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-

Annex ZA
(normative)**Normative references to international publications
with their corresponding European publications**

The following referenced documents are indispensable for the application 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.

NOTE Where an international publication has been modified by common modifications, indicated by (mod), the relevant EN/HD applies.

<u>Publication</u>	<u>Year</u>	<u>Title</u>	<u>EN/HD</u>	<u>Year</u>
IEC 60050-815	2000	International Electrotechnical Vocabulary (IEV) Chapter 815: Superconductivity	-	-

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