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Part 16: Electric characteristic measurements — Power-dependent surface resistance of superconductors at microwave frequencies

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Partie 16: Mesures de caractéristiques électroniques -
Résistance de surface des supraconducteurs aux hyperfréquences en fonction de la puissance
(CEI 61788-16:2013)

Supraleitfähigkeit -
Teil 16: Messung der elektronischen Eigenschaften -
Leistungsabhängiger Oberflächenwiderstand bei Mikrowellenfrequenzen
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Foreword

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Superconductivity - Part 15: Electronic characteristic measurements - Intrinsic surface impedance of superconductor films at microwave frequencies	EN 61788-15	-

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INTRODUCTION

Since the discovery of high-T_c superconductors (HTS), extensive researches have been performed worldwide for electronic applications and large-scale applications.

In the fields of electronics, especially in telecommunications, microwave passive devices such as filters using HTS are being developed and testing is underway on sites [1,2,3,4][1].

Superconductor materials for microwave resonators, filters, antennas and delay lines have the advantage of ultra-low loss characteristics. Knowledge of this parameter is vital for the development of new materials on the supplier side and the design of superconductor microwave components on the customer side. The parameters of superconductor materials needed to design microwave components are the surface resistance R_s and the temperature dependence of the R_s. Recent advances in HTS thin films with R_s, several orders of magnitude lower than normal metals has increased the need for a reliable characterization technique to measure this property [5,6]. Among several methods to measure the R_s of superconductor materials at microwave frequencies, the dielectric resonator method [7,8,9] has been useful due to that the method enables to measure the R_s nondestructively and accurately. In particular, the sapphire resonator is an excellent tool for measuring the R_s of HTS materials [10]. In 2002, the International Electrotechnical Commission (IEC) published the dielectric resonator method as a measurement standard [11].

The test method given in this standard enables measurement of the power-dependent surface resistance of superconductors at microwave frequencies. For high power microwave device applications such as those of transmitting devices, not only the temperature dependence of R_s but also the power dependence of R_s is needed to design the microwave components. Based on the measured power dependence, the RF current density dependence of the surface resistance can be evaluated. The simulation software to design the device gives the RF current distribution in the device. The results of the power dependence measurement can be directly compared with the simulation and allow the power handling capability of the device to be evaluated.

The test method given in this standard can be also applied to other superconductor bulk plates including low-T_c material.

This standard is intended to give an appropriate and agreeable technical base for the time being to those engineers working in the fields of electronics and superconductivity technology.

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

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[1] Numbers in square brackets refer to the Bibliography.

SUPERCONDUCTIVITY –

Part 16: Electronic characteristic measurements – Power-dependent surface resistance of superconductors at microwave frequencies

1 Scope

This part of IEC 61788 involves describing the standard measurement method of power-dependent surface resistance of superconductors at microwave frequencies by the sapphire resonator method. The measuring item is the power dependence of R_s at the resonant frequency.

The following is the applicable measuring range of surface resistances for this method:

Frequency: $f \sim 10$ GHz

Input microwave power: $P_{in} < 37$ dBm (5 W)

The aim is to report the surface resistance data at the measured frequency and that scaled to 10 GHz using the $R_s \propto f^2$ relation for comparison.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60050 (all parts), *International Electrotechnical Vocabulary* (available at: <http://www.electropedia.com>)

IEC 61788-15, *Superconductivity – Part 15: Electronic characteristic measurements – Intrinsic surface impedance of superconductor films at microwave frequencies*

3 Terms and definitions

For the purposes of this document, the definitions given in IEC 60050-815, one of which is repeated here for convenience, apply.

3.1
surface impedance
impedance of a material for a high frequency electromagnetic wave which is constrained to the surface of the material in the case of metals and superconductors

Note 1 to entry: The surface impedance governs the thermal losses of superconducting RF cavities.

Note 2 to entry: In general, surface impedance Z_s for conductors including superconductors is defined as the ratio of the electric field E_t to the magnetic field H_t, tangential to a conductor surface:

$$Z_s = E_t / H_t = R_s + jX_s,$$

where R_s is the surface resistance and X_s is the surface reactance.

4 Requirements

The surface resistance R_s of a superconductor film shall be measured by applying a microwave signal to a sapphire resonator with the superconductor film specimen and then measuring the insertion attenuation of the resonator at each frequency. The frequency shall be swept around the resonant frequency as the center and the insertion attenuation - frequency characteristics shall be recorded to obtain the *Q*-value, which corresponds to the loss.

The target relative combined standard uncertainty of this method is the coefficient of variation (standard deviation divided by the average of the surface resistance determinations), which is less than 20 % for a measurement temperature range from 30 K to 80 K.

It is the responsibility of the user of this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Hazards exist in such measurement. The use of a cryogenic system is essential to cool the superconductors and allow transition into the superconducting state. Direct contact of skin with cold apparatus components can cause immediate freezing, as can direct contact with a spilled cryogen. The use of an RF-generator is also essential to measure the high-frequency properties of materials. If its power is excessive, direct contact to human bodies could cause immediate burns.

5 Apparatus

5.1 Measurement system

5.1.1 Measurement system for the tan δ of the sapphire rod

Figure 1 shows a schematic diagram of the system required for the tan δ measurement. The system consists of a network analyzer system for transmission measurements, a measurement apparatus in which a sapphire resonator with superconductor films is fixed, and a thermometer for monitoring the measuring temperature.

The incident power generated from a suitable microwave source such as a synthesized sweeper is applied to the sapphire resonator fixed in the measurement apparatus. The transmission characteristics are shown on the display of the network analyzer. The measurement apparatus is fixed in a temperature-controlled cryocooler.

To measure the tan δ of the sapphire rod, a vector network analyzer is recommended, since its measurement accuracy is superior to a scalar network analyzer due to its wide dynamic range.

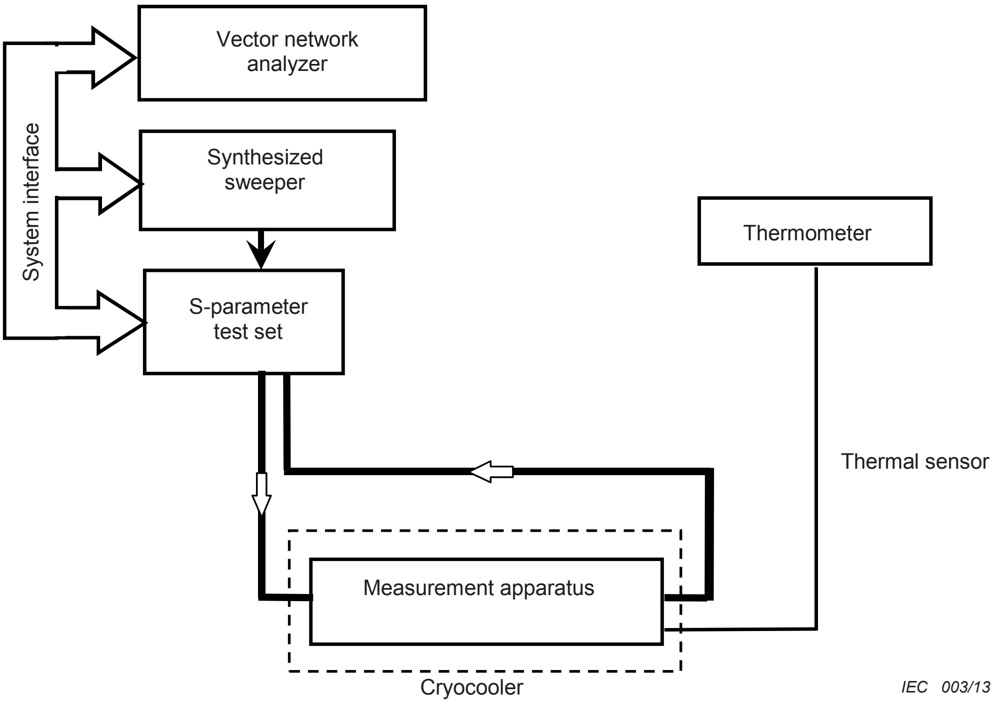


Figure 1 – Measurement system for tan δ of the sapphire rod

5.1.2 Measurement system for the power dependence of the surface resistance of superconductors at microwave frequencies

Figure 2 shows the measurement system for the power dependence of the surface resistance of superconductors using a sapphire resonator. A travelling wave tube (TWT) power amplifier with a maximum output power of around 40 dBm is inserted at the input into the resonator. The maximum input power into the resonator is around 37 dBm in this measurement system shown in Figure 2. The typical maximum input power of a network analyzer is in the order of 0 dBm, so a measurement circuit shall be designed to avoid direct exposure of high powered microwaves to the network analyzer, and also by using a circulator and an attenuator, significant reflection from the sapphire resonator should not affect the TWT amplifier.

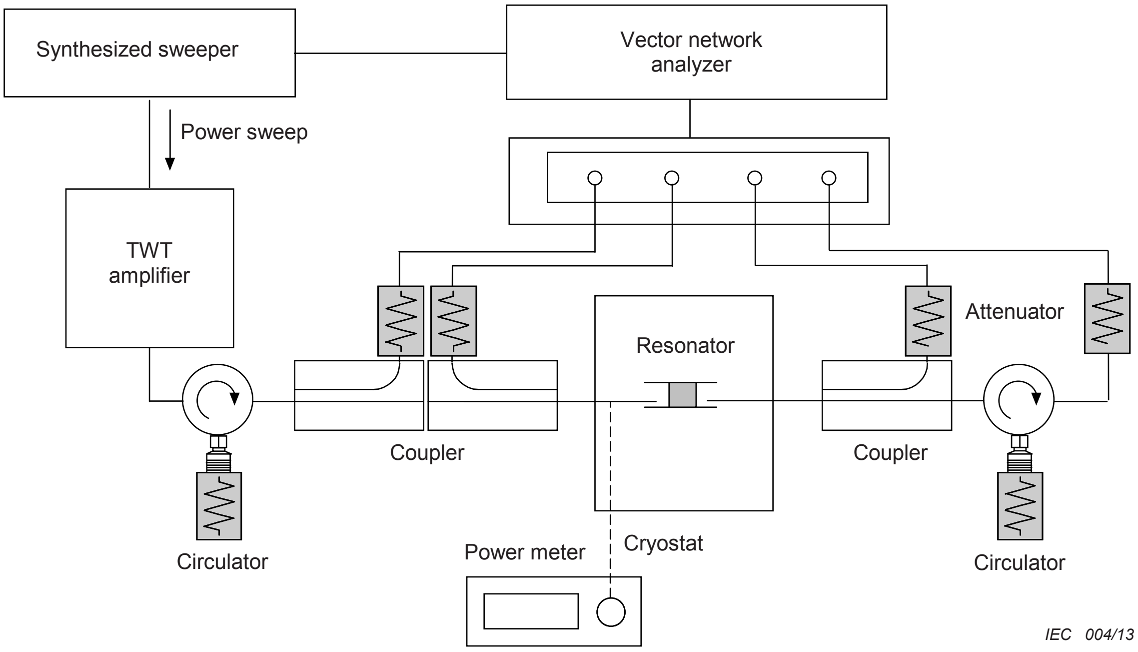


Figure 2 – Measurement system for the microwave power dependence of the surface resistance

Incident microwave power to the resonator is calibrated using a power meter before the measurement (dotted line in Figure 2). The incident power of the microwave is swept by changing the input power of the TWT amplifier.

5.2 Measurement apparatus

5.2.1 Sapphire resonator

Figure 3 shows a schematic diagram of a typical sapphire resonator (open type resonator) used to measure R_s of superconductor films and tan δ of the sapphire rod [9]. In the sapphire resonator, a sapphire rod was sandwiched between two superconducting films. The upper superconductor film is pressed down by a spring, which is made of phosphor bronze. The use of a plate type spring is recommended to improve measurement accuracy. This type of spring reduces the friction between the spring and the rest of the apparatus, and facilitates the movement of superconductor films during the thermal expansion of the sapphire rod.

Two semi-rigid cables for measuring transmission characteristics of the resonator shall be attached on both sides of the resonator in axially symmetrical positions ($\phi = 0$ and π, where ϕ is the rotational angle around the central axis of the sapphire rod). A semi-rigid cable with an outer diameter of 3,50 mm is recommended. Each of the two semi-rigid cables shall have a small loop at the end. The plane of the loop shall be set parallel to that of the superconductor films in order to suppress the unwanted TM_{mn0} modes. The coupling loops shall be carefully checked for cracks in the spot weld joint that may have developed upon repeated thermal cycling. These cables can move right and left to adjust the insertion attenuation (IA). In this adjustment, coupling of unwanted modes to the interested resonance mode shall be suppressed. Unwanted coupling to the other modes reduces the high *Q* value of the TE mode resonator. To suppress the unwanted coupling, special attention shall be paid to designing high *Q* resonators. Two other types of resonators usable along with the open type shown in Figure 3 are explained in A.1.

A reference line made of a semi-rigid cable shall be used to measure the full transmission power level, i.e. the reference level. The cable length equals to the sum of the two cables of the measurement apparatus.

To minimize the measurement error, two superconductor films shall be set in parallel. To ensure that the two superconductor films remain in tight contact with the ends of the sapphire rod, without any air gap, the surface of the two films and both ends of the rod shall be cleaned carefully.

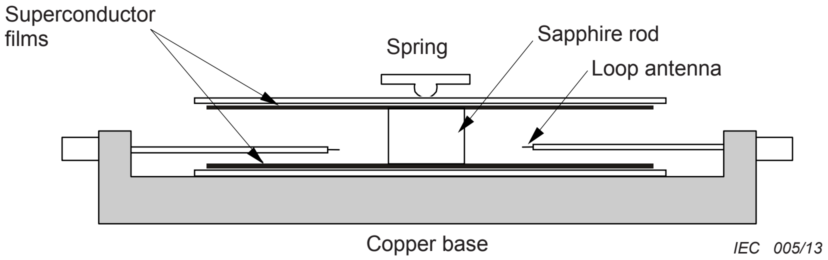


Figure 3 – Sapphire resonator (open type) to measure the surface resistance of superconductor films

5.2.2 Sapphire rod

A high-quality sapphire rod with low tan δ is required to achieve the requisite measurement accuracy on R_s. A recommended sapphire rod is expected to have a tan δ less than 10^{-6} at 77 K. To minimize the measurement error in R_s of the superconductor films, both ends of the sapphire rods shall be polished parallel to each other and perpendicular to the axis. Specifications of the sapphire rods are described in 7.1.

The diameter and height of the sapphire rod shall be carefully designed to ensure the TE_{011}, TE_{021} and TE_{012} modes do not couple to other TM, HE and EH modes, since coupling between TE mode and other modes causes the unloaded Q to deteriorate. The design guideline for the sapphire rod is described in A.2. Table 1 shows typical dimensions of the sapphire rod for a TE_{011}–mode resonant frequency of about 10 GHz.

Table 1 – Typical dimensions of the sapphire rod

Resonance Mode	**Frequency** GHz	**Diameter**	**Height**
		d mm	h mm
TE_{011}	10,6	11,8	6,74
TE_{021}	17,0		
TE_{012}	17,0		

5.2.3 Superconductor films

The diameter of the superconductor films shall be about three times larger than that of the sapphire rods. In this configuration, the increased uncertainty of R_s due to the radiation loss can be considered negligible, given the target relative combined standard uncertainty of 20%.

The film thickness shall be more than three times larger than the London penetration depth value at each temperature. If the film thickness is less than three times the London penetration depth, the measured R_s should mean the effective surface resistance.

6 Measurement procedure

6.1 Set-up

All the components of the sapphire resonator, such as the sapphire rod, superconductor films, and so on, shall be kept in a clean and dry state such as in a dry box or desiccator, as high humidity may degrade the unloaded Q-value.

The sapphire resonator shall be fixed in a specimen chamber inside the temperature-controlled cryocooler. The specimen chamber shall be generally evacuated. The temperatures of the superconductor films and sapphire rod shall be measured by a diode thermometer, or a thermocouple. The temperatures of the upper and lower superconductor films, and the sapphire rod must be kept as close as possible. This can be achieved by covering the sapphire resonator with aluminum foil, or filling the specimen chamber with helium gas.

6.2 Measurement of the tan δ of the sapphire rod

6.2.1 General

To measure the surface resistance of the superconductor films precisely using a sapphire resonator, the tan δ of the sapphire rod shall be known. The two-resonance mode dielectric resonator method [12,13], which uses the TE_{021} and TE_{012} modes of the same sapphire resonator shall be adopted to measure the tan δ of the sapphire rod. The measurement procedure of the tan δ is as follows:

6.2.2 Measurement of the frequency response of the TE_{021} mode

The temperature dependence of the resonant frequency f_0 and unloaded quality factor Q_u for TE_{021} resonance mode shall be measured as follows:

a) Connect the measurement system as shown in Figure 1. Fix the distance between the sapphire rod and each of the loops of the semi-rigid cables to be equal, so that this transmission-type resonator can be under-coupled equally to both loops. The coupling shall be adjusted to be weak enough not to excite unwanted resonance modes such as TM, HE and EH modes but strong enough to be able to excite TE_{021} mode. The input power to the resonator shall be below 10 dBm (typically 0 dBm). Confirm that the insertion attenuation of this mode is larger than 20 dB from the reference level. Evacuate and cool down the specimen chamber to below the critical temperature.

b) Measure S_{21} as a function of frequency where S_{21} is the transmission scattering parameter. Find the TE_{021} mode $|S_{21}|$ resonance peak of this resonator at a frequency nearly equal to the designed value of the resonant frequency f_0.

c) Narrow the frequency span on the display so that only the $|S_{21}|$ resonance peak of TE_{021} mode can be shown.

d) Collect both real and imaginary parts of the S_{21} , S_{11} and S_{22} as a function of frequency ($S_{21}(f)$, $S_{11}(f)$ and $S_{22}(f)$) where S_{11} and S_{22} are reflection scattering parameters.

e) Resonant frequency f_0 and loaded Q-value Q_L are obtained by fitting the experimentally measured data $S_{21}(f)$ to the Equation (1), where f_0 and Q_L are fitting parameters.

$$S_{21}(f) = \frac{S_{21}(f_0)}{1 + \mathrm{j}Q_\mathrm{L}\Delta(f)} \tag{1}$$

where f is frequency and $\Delta(f)$ is defined as

$$\Delta(f) = 1 - \frac{f_0^2}{f^2} \tag{2}$$

This fitting technique is called the “Circle fit technique”, the details of which are described in A.3.

f) The unloaded Q-value, Q_U, shall be extracted from the Q_L by the following Equation (3):

$$Q_\mathrm{U} = Q_\mathrm{L}(1 + \beta_1 + \beta_2) \tag{3}$$

where β_1 and β_2 are the coupling coefficients and defined as

$$\beta_1 = \frac{1 - |S_{11}|}{|S_{11}| + |S_{22}|} \tag{4}$$

$$\beta_2 = \frac{1 - |S_{22}|}{|S_{11}| + |S_{22}|} \tag{5}$$

where $|S_{11}|$ and $|S_{22}|$ are dips in the reflection scattering parameters at f_0 as shown in Figure 4, and measured in linear units of power rather than relative dB.

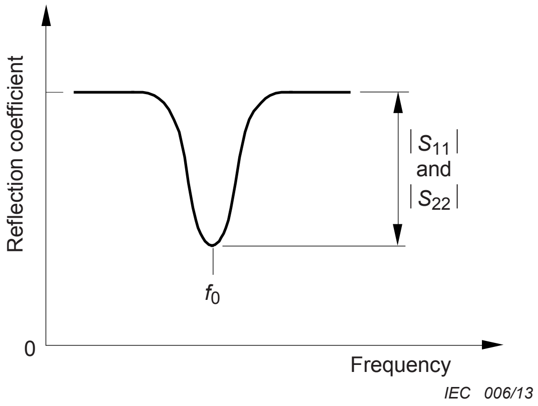


Figure 4 – Reflection scattering parameters ($|S_{11}|$ and $|S_{22}|$)

g) The f_0 and Q_U measured for this TE_{021} mode are denoted as f_{021} and Q_{U021}. By slowly changing the temperature of the cryocooler, the temperature dependence of f_{021} and Q_{U021} shall be measured.

6.2.3 Measurement of the frequency response of the TE_{012} mode

The temperature dependence of the resonant frequency f_0 and unloaded quality factor Q_U for the TE_{012} resonance mode shall be measured similarly to the TE_{021} resonance mode. The procedure is as follows:

a) After measuring the TE_{021} mode, cool down the specimen chamber below the critical temperature again.

b) Measure S_{21} as a function of frequency. Find the TE_{012} mode $|S_{21}|$ resonance peak of this resonator at a frequency nearly equal to the designed value of the resonant frequency f_0.

c) Narrow the frequency span on the display so that only the $|S_{21}|$ resonance peak of TE_{012} mode can be shown.

d) Follow step 6.2.2 d) to g) to measure the temperature dependence of the resonant frequency f_0 and the unloaded Q value Q_U for this TE_{012} mode. They are denoted as f_{012} and Q_{U012}.

6.2.4 Determination of tan δ of the sapphire rod

Using the measured value of f_{021}, Q_{U021}, f_{012} and Q_{U012}, the surface resistance of the superconductor films R_s and tan δ of the sapphire rod are given by the following simultaneous equations:

$$\left.\begin{aligned} R_s(f_{012}) &= \frac{1}{B_{012}}\left\{\frac{A_{012}}{Q_{U012}} - tan\delta(f_{012})\right\} \\ R_s(f_{021}) &= \frac{1}{B_{021}}\left\{\frac{A_{021}}{Q_{U021}} - tan\delta(f_{021})\right\} \end{aligned}\right\} \tag{6}$$

where A_{012}, B_{012}, A_{021} and B_{021} are geometric factors of TE_{012} and TE_{021}, respectively, and given by

$$A = 1 + \frac{W}{\varepsilon'} \tag{7}$$

$$B = p^2\left(\frac{\lambda_0}{2h}\right)^3 \frac{1+W}{30\pi^2\varepsilon'},\ \ p = 1,2,\cdots, \tag{8}$$

$$\lambda_0 = \frac{c}{f_0} \tag{9}$$

$$W = \frac{J_1^2(u)}{K_1^2(v)} \frac{K_0(v)K_2(v) - K_1^2(v)}{J_1^2(u) - J_0(u)J_2(u)} \tag{10}$$

$$v^2 = \left(\frac{\pi d}{\lambda_0}\right)^2 \left[\left(\frac{p\lambda_0}{2h}\right)^2 -1\right] \tag{11}$$

$$u\frac{J_0(u)}{J_1(u)} = -v\frac{K_0(v)}{K_1(v)} \tag{12}$$

where,

λ_0 is the free space resonant wavelength;

c is the velocity of light in a vacuum (c = 2,9979 × 10^8 m/s);

h is the height of the sapphire rod, and d is the diameter of the sapphire rod.

In the equations, $f_0 = f_{012}$ and p = 2 for TE_{012} mode, and $f_0 = f_{021}$ and p = 1 for TE_{021} mode, respectively.

The value u^2 is given by the transcendental Equation (12) using the value of v^2, where $J_n(u)$ is the Bessel function of the first kind and $K_n(v)$ is the modified Bessel function of the second kind, respectively. For any value of v, the m-th solution u exists between u_{0m} and u_{1m}, where $J_0(u_{0m}) = 0$ and $J_1(u_{1m}) = 0$. m = 1 for TE_{012} mode and m = 2 for TE_{021} mode.

In Equation (8), both R_s and tan δ are frequency-dependent and the scaling relations $R_s \propto f^2$ as explained by the two-fluid model, and tan $\delta \propto f$ an assumed relation for low-loss dielectrics, can be applied.

$$R_s(f_{021}) = R_s(f_{012}) \times (f_{021} / f_{012})^2 \tag{13}$$

$$\tan\delta(f_{021}) = \tan\delta(f_{012}) \times (f_{021} / f_{012}) \tag{14}$$

In Equations (7) and (8), ε' is the relative permittivity of the sapphire rod and given by

$$\varepsilon' = \left(\frac{\lambda_0}{\pi d}\right)^2 \left(u^2 + v^2\right) + 1 \tag{15}$$

using the values of v^2 and u^2.

6.3 Power dependence measurement

6.3.1 General

Once the tan δ of the sapphire rod has been measured, the surface resistance and its power dependence can be evaluated using the single resonance mode. TE_{011} is suitable for this measurement because of the strong resonance peak. The experimental procedure for the power dependence measurements is as follows.

6.3.2 Calibration of the incident microwave power to the resonator

The incident microwave power to the resonator shall be calibrated using a power meter before the measurement (dotted line in Figure 2). The incident power to the resonator, P_{in}, was determined as the measured power at the input of the resonator.

6.3.3 Measurement of the reference level

The level of full transmission power (reference level) shall be measured first. Connect the reference line of the semi-rigid cable between the input and output connectors. Subsequently, measure the transmission power level over the entire measurement frequency and temperature range. The reference level can change several decibels when the temperature of the apparatus changes from room temperature to the lowest measurement temperature. Therefore, the temperature dependence of the reference level must be taken into account.

6.3.4 Surface resistance measurement as a function of the incident microwave power

a) Connect the measurement system as shown in Figure 2. Fix the distance between the sapphire rod and the loops of the semi-rigid cables using a strong coupling, so that high microwave power can be introduced into the resonator. A suitable coupling strength is $|S_{11}| \cong 3$ dB. Cool down the specimen chamber to below the critical temperature.

b) Measure S_{21} as a function of frequency. Find the TE_{011} mode $|S_{21}|$ resonance peak of this resonator at a frequency nearly equal to the designed value of the resonant frequency f_0.

c) Narrow the frequency span on the display so that only the $|S_{21}|$ resonance peak of TE_{011} mode can be shown. Measure the insertion attenuation, a_{ins}, which is the attenuation (in dB) from the reference level to the $|S_{21}|$ at the resonant frequency f_0 of the TE_{011} mode.

d) Collect both real and imaginary parts of the S_{21} and S_{11} as a function of frequency ($S_{21}(f)$ and $S_{11}(f)$)

e) Follow the step 6.2.2 e) to measure the resonant frequency f_0 and the loaded Q value Q_{L} for this TE_{011} mode. They are denoted as f_{011} and $Q_{\mathrm{L}011}$.

f) Extract the unloaded Q value, $Q_{\mathrm{U}011}$, from the $Q_{\mathrm{L}011}$ by the following equation:

$$Q_{\mathrm{U}011} = \frac{Q_{\mathrm{L}011}}{1 - A_{\mathrm{t}}}, A_{\mathrm{t}} = 10^{-a_{\mathrm{ins}}/20} \tag{16}$$

g) The surface resistances of the superconductor films are obtained by the following equation:

$$R_{\mathrm{s}}(f_{011}) = \frac{1}{B_{011}} \left\{ \frac{A_{011}}{Q_{\mathrm{U}011}} - tan\delta(f_{011}) \right\} \tag{17}$$

where $A_{011}$and B_{011} are geometric factors of TE_{011} mode, and obtained by equations (7) to (15) setting $f_0 = f_{011}$, $p = 1$, and $m = 1$. The tan $\delta(f_{011})$ should be the scaled value at f_{011} of the value determined in 6.2.4 which corresponds to f_{012},

$$tan\,\delta(f_{011}) = tan\,\delta(f_{012}) \times (f_{011} / f_{012}) \tag{18}$$

h) The incident power of the microwave was swept by changing the input power of the TWT amplifier with the specimen chamber maintained at a constant temperature. Repeat steps c) to g) for each incident microwave power.

i) Change the temperature of the specimen chamber and repeat steps c) to h) for each temperature.

6.3.5 Determination of the maximum surface magnetic flux density

The measured incident microwave power dependence of the surface resistance itself does not directly show the power handling capability of the superconductor films. The latter shall be measured in terms of the maximum surface magnetic flux density without causing its properties to deteriorate. High surface magnetic flux density, i.e., RF current induces the pair breaking of the Cooper pair and increases the surface resistance. Also weak coupling between the grain boundaries or d-wave symmetry of the superconductor is considered to increase the surface resistance.

From the measured incident power dependence of the surface resistance, the maximum surface magnetic flux density shall be calculated as follows [14,15].

The dissipated power in the resonator P_0 is evaluated from the incident power to the resonator P_{in} and S parameters as follows:

$$P_0 = P_{\mathrm{in}}(1 - |S_{11}|^2 - |S_{21}|^2) \tag{19}$$

The surface magnetic flux density of the superconducting films can be calculated by the analytical equation. The maximum surface magnetic flux density $B_{\mathrm{s\,max}}$ is given by the following equation [14]:

$$B_{\mathrm{s\,max}} = 0{,}581865 \left\{ \frac{2\pi R_{\mathrm{s}}}{P_0} \int_0^{d/2} J_1^2\left(\frac{2u}{d}\rho\right)\rho d\rho \left[1 + W + \frac{240\pi^2 \varepsilon' \tan\delta}{R_{\mathrm{s}}} \left(\frac{h}{\lambda_0} \right)^3 \right] \right\}^{-1/2} \tag{20}$$

where d, J_1, u, W, ε', h and λ_0 are the same as defined in Equations (7) to (15), and λ_{d} is the penetration depth of the superconductor films. The λ_{d} can be directly measured according to IEC 61788-15. When the directly measured λ_{d} data is not available, a typical reported value for the same material should be used.

7 Uncertainty of the test method

7.1 Surface resistance

A vector network analyzer as specified in Table 2 shall be used to record the frequency dependence of attenuation. The resulting record shall allow the determination of Q to a relative uncertainty of 10^{-2}.

Table 2 – Specifications of the vector network analyzer

Dynamic range of S_{21}	above 60 dB
Frequency resolution	below 1 Hz
Attenuation uncertainty	below 0,1 dB
Input power limitation	below 10 dBm

The specifications of the sapphire rod are shown in Table 3. Term definitions in Table 3 are shown in Figure 5.

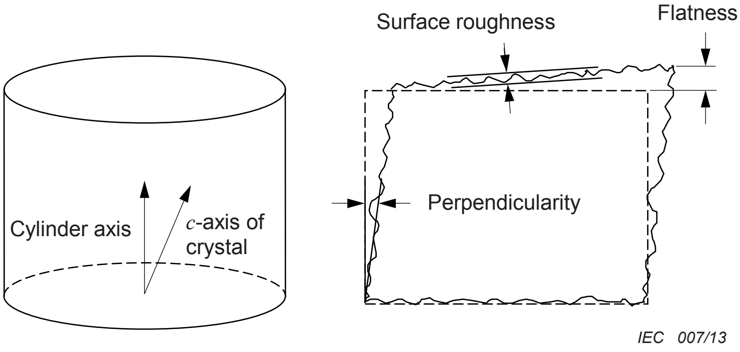


Figure 5 – Term definitions in Table 3

Table 3 – Specifications of the sapphire rods

Tolerance in diameter	±0,05 mm
Tolerance in height	±0,05 mm
Flatness	below 0,005 mm
Surface roughness	top and bottom surface: root mean square height below 10 nm cylindrical surface: root mean square height below 0,001 mm
Perpendicularity	within 0,1°
Axis	parallel to c-axis within 0,3°

7.2 Temperature

The measurement apparatus is cooled down to the specified temperature by any means during testing. An easy choice would be to immerse the apparatus into a liquid cryogen. This technique is quick and simple and yields a known and stable temperature. Unfortunately, most HTS materials are damaged by the condensation of moisture that occurs when removing the sample from the cryogen. In addition, uncertainties generated by the presence of a gas/liquid mixture within the cavity, and the inability to measure R_s as a function of temperature support the use of other cooling methods. These limitations can be circumvented by the immersion of a vacuum can into a liquid cryogen. If the vacuum can is backfilled with gas, then rapid cooling and uniform temperatures occur. If heaters are attached to the apparatus, then the temperature-dependent properties of the HTS material can be measured. A third and equally good choice is the use of a cryocooler. In this case, the resonator is under vacuum and cooled by conduction through the metallic package. Care must be taken to avoid temperature gradients with the apparatus.

A cryostat shall be provided with the necessary environment for measuring R_s and the specimen shall be measured while in a stable and isothermal state. The specimen temperature is assumed to be the same as that of the sample holder. The holder temperature shall be reported to an accuracy of ±2 K, measured using an appropriate temperature sensor.

The difference between the specimen and holder temperatures shall be minimized by using shields with good thermal conductivity.

For power dependence measurement, heating of the loop antenna by elevated microwave power level may affect the measurements. To minimize the heating effects, the distance between the sapphire rod and the loops of the semi-rigid cables should be short enough to realize a strong coupling and to reduce the incident microwave power level for the power measurement. A suitable coupling strength is $|S_{11}| \cong 3$ dB, as shown in 6.3.4 a).

7.3 Specimen and holder support structure

The support structure shall provide adequate support for the specimen. It is imperative that the two films be parallel and mechanically stable throughout the measurement, especially in a cryocooler and over a wide temperature range.

7.4 Specimen protection

Condensation of moisture and scratching of the film deteriorate superconducting properties. Some protection measures should be provided for the specimens. Polytetrafluoroethylene (PTFE) or Polymethylmethacrylate (PMMA) coating does not affect the measurements, thus they can be used for protection [16]. A coating material thickness of less than several micrometers is recommended.

8 Test report

8.1 Identification of the test specimen

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

a) name of the manufacturer of the specimen;
b) classification and/or symbol;
c) lot number;
d) chemical compositions of the thin film and substrate;
e) thickness and roughness of the thin film;
f) manufacturing process technique.

8.2 Report of power dependence of R_s values

The R_s values, along with their corresponding f_{011}, Q_u, ε', tan δ, P_{in} values, and their maximum surface magnetic flux density ($B_{s\ max}$) dependence shall be reported.

8.3 Report of test conditions

The following test conditions shall be reported:

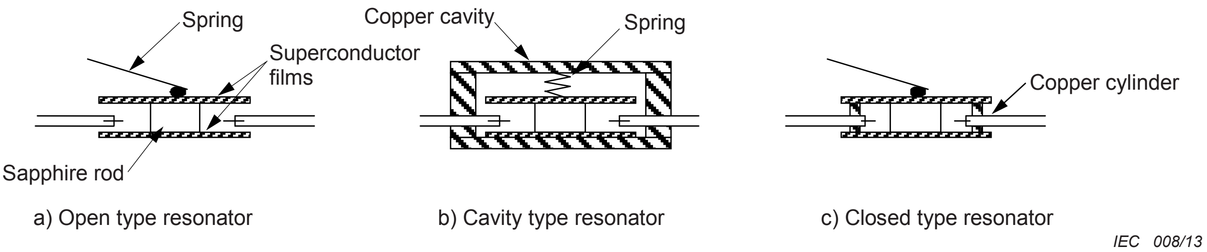
a) test frequency and resolution of frequency;
b) test maximum RF incident power;
c) test temperature, uncertainty of temperature and temperature difference of two plates;
d) history of sample temperature versus time.

Annex A
(informative)

Additional information relating to Clauses 1 to 7

A.1 Three types of sapphire rod resonators

Unwanted parasitic coupling to the other mode reduces the high *Q*-value of the TE mode resonator. To suppress the parasitic coupling, special attention is paid to design high *Q* resonators. Three types of resonators are proposed and shown in Figure A.1:



a) Open type resonator b) Cavity type resonator c) Closed type resonator

Figure A.1 – Three types of sapphire rod resonators

a) Open type resonator: a low loss sapphire rod is placed between two parallel superconductor films. Two semi-rigid cables for the RF input and output magnetic dipole coupling are attached on both sides of the resonator. In this configuration, the vertical position of the coupling cables should be carefully designed so as to prevent the radiation loss from propagating along the coupling cables, which degrades the high *Q* of the TE_{0mp} mode and causes increased error for the R_s measurements.

b) Cavity type resonator: the open type resonator shown in a) is placed inside a conductor (copper) cavity.

c) Closed type resonator: a conductor (copper) cylinder is put between the superconductor films. In this configuration, the radiation loss along the coupling cable is strongly blocked by the copper cylinder.

The measuring apparatus on the cryocooler is protected from mechanical and thermal disturbances, e.g. by using vibration absorbers and/or by covering the apparatus with radiation shield, and installed in an *X*-*Y* and/or *Z*-axial manipulator for adjusting sample positions within the range of approximately ±1 mm.

A loop length of the antenna is designed on the basis of the quarter wavelength rule to achieve the maximum measuring sensitivity.

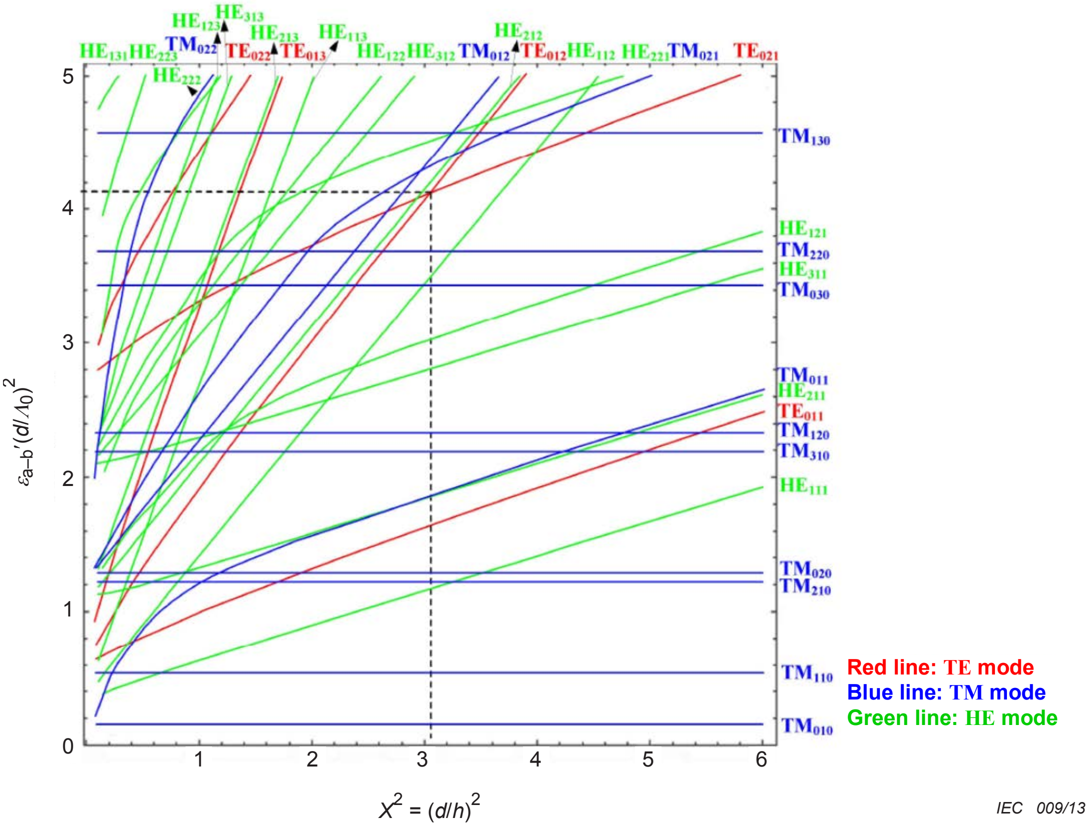
A.2 Dimensions of the sapphire rod

The two-resonance mode dielectric resonator method used in this standard uses a single sapphire resonator that differs from the existing IEC standard (IEC 61788-7:2006) which uses two sapphire resonators with nearly the same tan δ quality. Use of a single sapphire resonator makes it possible to reduce uncertainty in the measured surface resistance that might result from using two sapphire resonators with sapphire rods of even slightly different quality.

The two-resonance mode dielectric resonator method uses the two modes of the same sapphire resonator, namely, TE_{012} and TE_{021} [1][2]. The sapphire rod is designed with these two modes located within a narrow frequency range, but not affecting each other. Also the coupling between these TE modes and other TM, HE and EH modes should be avoided.

Figure A.2 shows the mode charts for designing the sapphire resonator used for the two-resonance mode dielectric resonator method, in which the uniaxial-anisotropic characteristics of the relative permittivity of the sapphire rod are taken into consideration (see IEC 61788-15). $\varepsilon_{a\text{-}b}$‘ is the relative permittivity in the plane perpendicular to the c-axis, d is the diameter of the sapphire rod, h is the height of the sapphire rod, and Λ_0 is the free space resonant wavelength.

As shown in Figure A.2, the value $(d/h)^2$ should be selected around 3,06 to ensure the TE_{012} and TE_{021} resonances are located close enough to each other and are not affected by the other modes.



IEC 009/13

NOTE The dotted line corresponds to the dimensions of the sapphire rod used for the two-resonance mode dielectric resonator method. Λ_0 denotes the wavelength in free space corresponding to the resonant frequency f_0 and $\Lambda_0 = c/f_0$ with $c = 2{,}9979 \times 10^8$ m/s. $\varepsilon_{a\text{-}b}' = 9{,}28$ and $\varepsilon_c' = 11{,}3$ are used in preparing this mode chart.

Figure A.2 – Mode chart for a sapphire resonator (see IEC 61788-15)

As the resonant frequency of TE mode is a function of relative permittivity and the dimensions of the sapphire rod, its diameter and height are selected so that the desired f_0 is obtained.

From the curve of the TE_{012} mode in Figure A.2, the value of $\varepsilon_{a\text{-}b}$‘ $(d/\Lambda_0)^2$ can be determined for each $(d/h)^2$ value. When the value $(d/h)^2$ equals 3,06, for example, the value of $\varepsilon_{a\text{-}b}$‘ $(d/\Lambda_0)^2$ equals 4,15. Thus, the resonant frequency of TE_{012} mode for the sapphire rod with dimension of

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[2] Figures in square brackets refer to the reference documents in A.5 of this annex.

$(d/h)^2 = 3{,}06$ is calculated from the following equation by specifying d and $\varepsilon_{a\text{-}b}'$ of the sapphire rod:

$$\varepsilon_{a\text{-}b}'(d/\Lambda_0)^2 = \varepsilon_{a\text{-}b}'(d \times f_0/c)^2 = 4{,}15 \tag{A.1}$$

For the two-resonance mode dielectric resonator measurement, the sapphire rod is designed to be 11,8 mm in diameter and 6,74 mm in height, and thus f_0 for either mode TE_{012} or TE_{021} is around 17 GHz. For the power-dependence measurement, f_0 for the TE_{011} mode is 10,6 GHz.

A.3 Circle fit technique

In principle, the accuracy of an R_s measurement and/or tan δ measurement mainly depends on that of the quality factor measurement. The circle fit technique can precisely measure Q_L. Figure A.3 shows a schematic of two methods used for Q_L measurements, namely, the conventional 3 dB method and the circle fit method.

The 3 dB method is widely used due to its simplicity. In the 3 dB method, Q_L is given by

$$Q_L = \frac{f_0}{\Delta f} \tag{A.2}$$

where f_0 is the resonant frequency and Δf is the half power band width ($\Delta f = f_2 - f_1$).

Most vector network analyzers have an automatic function that measures Q_L by using the 3 dB method. However, this method uses only three points of the resonance peak and assumes an ideal symmetric resonance peak. Actual resonance peaks frequently exhibit asymmetric shapes due to the unwanted mode coupling effect. Moreover, when the coupling is very weak, measuring Q_L is difficult due to noise in the data.

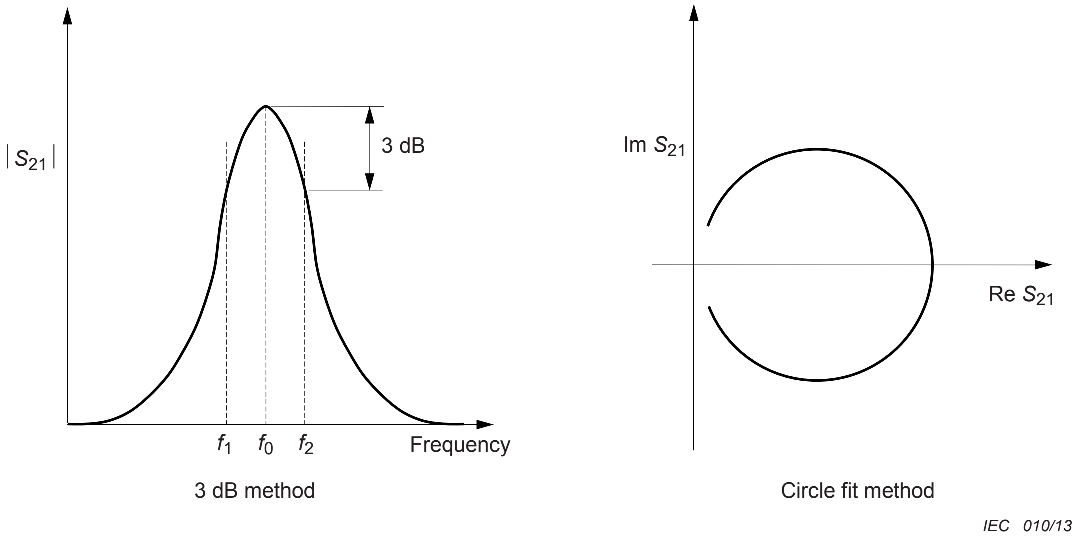

Figure A.3 – Loaded quality factor Q_L measurements using the conventional 3 dB method and the circle fit method

The circle fit technique [2] is suitable for Q_L measurement when the resonance has an unwanted mode or very weak couplings. Figure A.3 shows the circle in the complex plane of S_{21}. For a simple equivalent circuit for the resonator, S_{21} can be defined as

$$S_{21}(f) = \frac{S_{21}(f_0)}{1 + \mathrm{j}Q_{\mathrm{L}}\Delta(f)} \tag{A.3}$$

where f is frequency, f_0 is resonance frequency, and $\Delta(f)$ is defined as

$$\Delta(f) = 1 - \frac{f_0^2}{f^2} \tag{A.4}$$

For numerical calculations, it is convenient to plot the f dependence of phase of S_{21}, $\varphi_{21}(f)$:

$$\phi_{21}(f) = -tan^{-1}(Q_{\mathrm{L}}\Delta(f)) \tag{A.5}$$

Q_{L} is calculated as the constant of Equation A.5. A proper frequency range for the fitting is nearly equal to that for the 3 dB method (from around f_1 to f_2). Using these fitting processes, many data points of f dependence of S_{21} are used, significantly improving measurement accuracy, especially when the resonance peak is very weak. Moreover, the circle fitting technique uses data in the complex plane and can exclude the effect of unwanted mode coupling.

A.4 Test results

Figure A.4 shows the measured tan δ of a sapphire rod designed for the two-resonance mode dielectric resonator method. Data was measured at 17 GHz and scaled to 10,7 GHz. The tan δ was in the order of 10^{-7}, and showed a slight increase with increasing temperature. The subsequent rapid decrease in tan δ was due to the ambiguity of the measured Q_{U} near T_{c} caused by the rapid change in Q_{U}. In the two-resonance mode dielectric resonator measurement, the temperature of the resonator must be scanned twice, and the resulting small difference in these two temperatures and consequently in the Q_{U} measurement has a significant effect near T_{c}. The rapid decrease in tan δ is not essential and does not reflect an intrinsic loss in the sapphire rod.

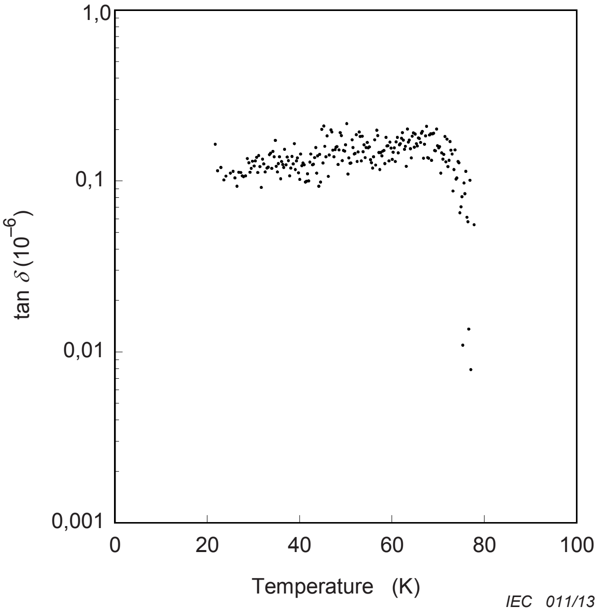

Figure A.4 – Temperature dependence of tan δ of a sapphire rod measured using the two-resonance mode dielectric resonator method [3]

Figure A.5 shows the maximum surface magnetic flux density dependence of R_s calculated from the measured input-power dependence of Q_u for two commercial YBCO films on MgO(100) substrates as an example.

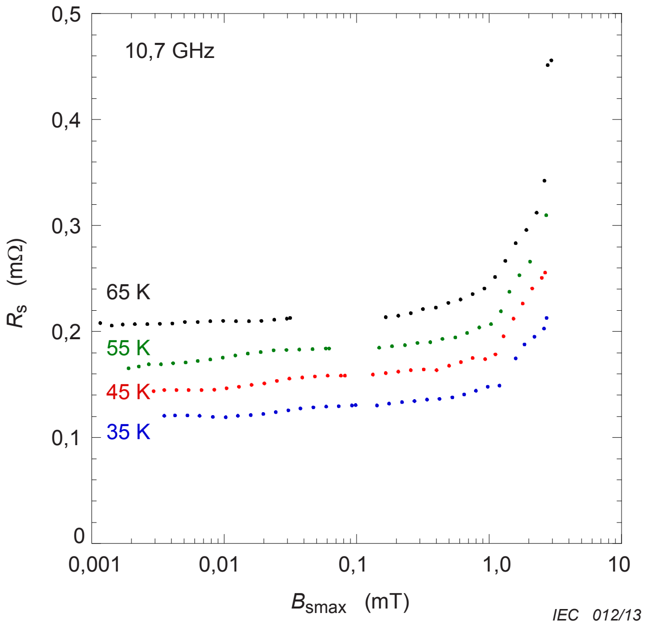

Figure A.5 – Dependence of the surface resistance R_s on the maximum surface magnetic flux density $B_{s\ max}$ [3]

A.5 Reference documents

[1] HASHIMOTO, T. and KOBAYASHI, Y. An image-type dielectric resonator method to measure surface resistance of a high-T_c superconductor film. *IEICE Trans. Electron.,* 2004, E87C No. 5, p. 681.

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Annex B
(informative)

Uncertainty considerations

B.1 Overview

In 1995, a number of international standards organizations, including IEC, decided to unify the use of statistical terms in their standards. It was decided to use the word “uncertainty” for all quantitative (associated with a number) statistical expressions and eliminate the quantitative use of “precision” and “accuracy.” The words “accuracy” and “precision” could still be used qualitatively. The terminology and methods of uncertainty evaluation are standardized in the Guide to the Expression of Uncertainty in Measurement (GUM) [1] [3].

It was left to each technical committee to decide if they were going to change existing and future standards to be consistent with the new unified approach. Such change is not easy and creates additional confusion, especially for those who are not familiar with statistics and the term uncertainty. At the June 2006 TC 90 meeting in Kyoto, it was decided to implement these changes in future standards.

Converting “accuracy” and “precision” numbers to the equivalent “uncertainty” numbers requires knowledge about the origins of the numbers. The coverage factor of the original number may have been 1, 2, 3, or some other number. A manufacturer’s specification that can sometimes be described by a rectangular distribution will lead to a conversion number of $1/\sqrt{3}$. The appropriate coverage factor was used when converting the original number to the equivalent standard uncertainty. The conversion process is not something that the user of the standard needs to address for compliance to TC 90 standards, it is only explained here to inform the user about how the numbers were changed in this process. The process of converting to uncertainty terminology does not alter the user’s need to evaluate their measurement uncertainty to determine if the criteria of the standard are met.

The procedures outlined in TC 90 measurement standards were designed to limit the uncertainty of any quantity that could influence the measurement, based on the Convener’s engineering judgment and propagation of error analysis. Where possible, the standards have simple limits for the influence of some quantities so that the user is not required to evaluate the uncertainty of such quantities. The overall uncertainty of a standard was then confirmed by an interlaboratory comparison.

B.2 Definitions

Statistical definitions can be found in three sources: the GUM, the International Vocabulary of Basic and General Terms in Metrology (VIM)[2], and the NIST Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results (NIST)[3]. Not all statistical terms used in this standard are explicitly defined in the GUM. For example, the terms “relative standard uncertainty” and “relative combined standard uncertainty” are used in the GUM:2008 (5.1.6, Annex J), but they are not formally defined in the GUM (see [3]).

B.3 Consideration of the uncertainty concept

Statistical evaluations in the past frequently used the coefficient of variation (COV) which is the ratio of the standard deviation and the mean (N.B. the COV is often called the relative standard deviation). Such evaluations have been used to assess the precision of the measurements and

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[3] Figures in square brackets refer to the reference documents in B.5 of this annex.

give the closeness of repeated tests. The standard uncertainty (SU) depends more on the number of repeated tests and less on the mean than the COV and therefore in some cases gives a more realistic picture of the data scatter and test judgment. The example below shows a set of electronic drift and creep voltage measurements from two nominally identical extensometers using same signal conditioner and data acquisition system. The n = 10 data pairs are taken randomly from the spreadsheet of 32 000 cells. Here, extensometer number one (E_1) is at zero offset position whilst extensometer number two (E_2) is deflected to 1 mm. The output signals are in volts.

Table B.1 – Output signals from two nominally identical extensometers

Output signal V	
E_1	E_2
0,00122070	2,33459473
0,00061035	2,33428955
0,00152588	2,33428955
0,00122070	2,33459473
0,00152588	2,33459473
0,00122070	2,33398438
0,00152588	2,33428955
0,00091553	2,33428955
0,00091553	2,33459473
0,00122070	2,33459473

Table B.2 – Mean values of two output signals

Mean ($\overline{X}$) V	
E_1	E_2
0,00119019	2,33441162

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n} \qquad [\mathrm{V}] \tag{B.1}$$

Table B.3 – Experimental standard deviations of two output signals

Experimental standard deviation (s) V	
E_1	E_2
0,00030348	0,000213381

$$s = \sqrt{\frac{1}{n-1} \cdot \sum_{i=1}^{n} \left(X_i - \overline{X}\right)^2} \qquad [\mathrm{V}] \tag{B.2}$$

Table B.4 – Standard uncertainties of two output signals

Standard uncertainty (u) V	
E_1	E_2
0,00009597	0,00006748

$$u = \frac{s}{\sqrt{n}} \quad [\mathrm{V}] \tag{B.3}$$

Table B.5 – Coefficient of Variations of two output signals

Coefficient of Variation (COV) %	
E_1	E_2
25,4982	0,0091

$$COV = \frac{s}{\overline{X}} \tag{B.4}$$

The standard uncertainty is very similar for the two extensometer deflections. In contrast the coefficient of variation *COV* is nearly a factor of 2800 different between the two data sets. This shows the advantage of using the standard uncertainty which is independent of the mean value.

B.4 Uncertainty evaluation example for TC 90 standards

The observed value of a measurement does not usually coincide with the true value of the measurand. The observed value may be considered as an estimate of the true value. The uncertainty is part of the "measurement error" which is an intrinsic part of any measurement. The magnitude of the uncertainty is both a measure of the metrological quality of the measurements and improves the knowledge about the measurement procedure. The result of any physical measurement consists of two parts: an estimate of the true value of the measurand and the uncertainty of this “best” estimate. The GUM, within this context, is a guide for a transparent, standardized documentation of the measurement procedure. One can attempt to measure the true value by measuring “the best estimate” and using uncertainty evaluations which can be considered as two types: Type A uncertainties (repeated measurements in the laboratory in general expressed in the form of Gaussian distributions) and Type B uncertainties (previous experiments, literature data, manufacturer’s information, etc. often provided in the form of rectangular distributions).

The calculation of uncertainty using the GUM procedure is illustrated in the following example:

a) The user must derive in the first step a mathematical measurement model in the form of identified measurand as a function of all input quantities. A simple example of such model is given for the uncertainty of a force, F_{LC} measurement using a load cell:

   $F_{\mathrm{LC}} = W + d_{\mathrm{w}} + d_{\mathrm{R}} + d_{\mathrm{Re}}$

   where W, d_{w}, d_{R}, and d_{Re} represent the weight of standard as expected, the manufacturer’s data, repeated checks of standard weight/day and the reproducibility of checks at different days, respectively.

   Here the input quantities are: the measured weight of standard weights using different balances (Type A), manufacturer’s data (Type B), repeated test results using the digital electronic system (Type B), and reproducibility of the final values measured on different days (Type B).

b) The user should identify the type of distribution for each input quantity (e.g. Gaussian distributions for Type A measurements and rectangular distributions for Type B measurements).

c) Evaluate the standard uncertainty of the Type A measurements,

   $u_A = \frac{s}{\sqrt{n}}$ where, s is the experimental standard deviation and n is the total number of measured data points.

d) Evaluate the standard uncertainties of the Type B measurements:

   $u_B = \sqrt{\frac{1}{3} \cdot {d_w}^2 + ...}$ where, d_w is the range of rectangular distributed values

e) Calculate the combined standard uncertainty for the measurand by combining all the standard uncertainties using the expression:

   $$u_c = \sqrt{u_A^2 + u_B^2}$$

   In this case, it has been assumed that there is no correlation between input quantities. If the model equation has terms with products or quotients, the combined standard uncertainty is evaluated using partial derivatives and the relationship becomes more complex due to the sensitivity coefficients [4, 5].

f) Optional - the combined standard uncertainty of the estimate of the referred measurand can be multiplied by a coverage factor (e. g. 1 for 68 % or 2 for 95 % or 3 for 99 %) to increase the probability that the measurand can be expected to lie within the interval.

g) Report the result as the estimate of the measurand ± the expanded uncertainty, together with the unit of measurement, and, at a minimum, state the coverage factor used to compute the expanded uncertainty and the estimated coverage probability.

To facilitate the computation and standardize the procedure, use of appropriate certified commercial software is a straightforward method that reduces the amount of routine work [6, 7]. In particular, the indicated partial derivatives can be easily obtained when such a software tool is used. Further references for the guidelines of measurement uncertainties are given in [3, 8, and 9].

B.5 Reference documents of Annex B

[1] ISO/IEC Guide 98-3:2008, *Uncertainty of measurement – Part 3: Guide to the expression of uncertainty in measurement* (GUM:1995)

[2] ISO/IEC Guide 99:2007, *International vocabulary of metrology – Basic and general concepts and associated terms (VIM)*

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