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Part 7:

Electronic characteristic measurements – Surface resistance of superconductors at microwave frequencies



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Part 7: Electronic characteristic measurements – Surface resistance of superconductors at microwave frequencies

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International Electrotechnical Commission, 3, rue de Varembé, PO Box 131, CH-1211 Geneva 20, Switzerland
Telephone: +41 22 919 02 11 Telefax: +41 22 919 03 00 E-mail: inmail@iec.ch Web: www.iec.ch



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CONTENTS

FOREWORD.....	4
INTRODUCTION.....	6
1 Scope.....	7
2 Normative references	7
3 Terms and definitions	7
4 Requirements.....	7
5 Apparatus.....	8
5.1 Measurement system	8
5.2 Measurement apparatus for R_s	9
5.3 Dielectric rods	11
6 Measurement procedure.....	12
6.1 Specimen preparation	12
6.2 Set-up	12
6.3 Measurement of reference level	12
6.4 Measurement of the frequency response of resonators.....	13
6.5 Determination of surface resistance of the superconductor and ε' and $\tan \delta$ of the standard sapphire rods.....	15
7 Precision and accuracy of the test method.....	16
7.1 Surface resistance	16
7.2 Temperature.....	17
7.3 Specimen and holder support structure	17
7.4 Specimen protection.....	18
8 Test report.....	18
8.1 Identification of test specimen	18
8.2 Report of R_s values.....	18
8.3 Report of test conditions.....	18
Annex A (informative) Additional information relating to Clauses 1 to 8.....	19
Bibliography.....	32
Figure 1 – Schematic diagram of measurement system for temperature dependence of R_s using a cryocooler	8
Figure 2 – Typical measurement apparatus for R_s	10
Figure 3 – Insertion attenuation IA , resonant frequency f_0 and half power bandwidth Δf , measured at T Kelvin	13
Figure 4 – Reflection scattering parameters (S_{11} and S_{22}).....	15
Figure 5 – Term definitions in Table 4.....	17
Figure A.1 – Schematic configuration of several measurement methods for the surface resistance	20
Figure A.2 – Configuration of a cylindrical dielectric rod resonator short-circuited at both ends by two parallel superconductor films deposited on dielectric substrates	22
Figure A.3 – Computed results of the u - v and W - v relations for TE_{01p} mode.....	23
Figure A.4 – Configuration of standard dielectric rods for measurement of R_s and $\tan \delta$	24

Figure A.5 – Three types of dielectric resonators	24
Figure A.6 – Mode chart to design TE_{011} resonator short-circuited at both ends by parallel superconductor films [11]	27
Figure A.7 – Mode chart to design TE_{013} resonator short-circuited at both ends by parallel superconductor films [11]	28
Figure A.8 – Mode chart for TE_{011} closed-type resonator.....	29
Figure A.9 – Mode chart for TE_{013} closed-type resonator.....	30
Table 1 – Typical dimensions of pairs of standard sapphire rods for 12 GHz, 18 GHz and 22 GHz	11
Table 2 – Dimensions of superconductor film for 12 GHz, 18 GHz, and 22 GHz.....	12
Table 3 – Specifications on vector network analyzer	16
Table 4 – Specifications on sapphire rods.....	16

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SUPERCONDUCTIVITY –**Part 7: Electronic characteristic measurements –
Surface resistance of superconductors
at microwave frequencies**

FOREWORD

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International Standard IEC 61788-7 has been prepared by IEC technical committee 90: Superconductivity.

This second edition cancels and replaces the first edition, published in 2002, of which it constitutes a technical revision. Examples of technical changes made are: 1) closed type resonators are recommended from the viewpoint of the stable measurements, 2) uniaxial-anisotropic characteristics of sapphire rods are taken into consideration for designing the size of the sapphire rods, and 3) recommended measurement frequency of 18 GHz and 22 GHz are added to 12 GHz described in the first edition.

The text of this standard is based on the following documents:

FDIS	Report on voting
90/193/FDIS	90/198/RVD

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

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

IEC 61788 consists of the following parts, under the general title *Superconductivity*:

- Part 1: Critical current measurement – DC critical current of Cu/Nb-Ti composite superconductors
- Part 2: Critical current measurement – DC critical current of Nb₃Sn composite superconductors
- Part 3: Critical current measurement – DC critical current of Ag- and/or Ag alloy-sheathed Bi-2212 and Bi-2223 oxide superconductors
- Part 4: Residual resistance ratio measurement – Residual resistance ratio of Nb-Ti composite superconductors
- Part 5: Matrix to superconductor volume ratio measurement – Copper to superconductor volume ratio of Cu/Nb-Ti composite superconductors
- Part 6: Mechanical properties measurement – Room temperature tensile test of Cu/Nb-Ti composite superconductors
- Part 7: Electronic characteristic measurements – Surface resistance of superconductors at microwave frequencies
- Part 8: AC loss measurements – Total AC loss measurement of Cu/Nb-Ti composite superconducting wires exposed to a transverse alternating magnetic field by a pickup coil method
- Part 9: Measurements for bulk high temperature superconductors – Trapped flux density of large grain oxide superconductors
- Part 10: Critical temperature measurement – Critical temperature of Nb-Ti, Nb₃Sn, and Bi-system oxide composite superconductors by a resistance method
- Part 11: Residual resistance ratio measurement – Residual resistance ratio of Nb₃Sn composite superconductors
- Part 12: Matrix to superconductor volume ratio measurement – Copper to non-copper volume ratio of Nb₃Sn composite superconducting wires
- Part 13: AC loss measurements – Magnetometer methods for hysteresis loss in Cu/Nb-Ti multifilamentary composites

The committee has decided that the contents of this publication will remain unchanged until the maintenance result date indicated on the IEC web site under "<http://webstore.iec.ch>" in the data related to the specific publication. At this date, the publication will be

- reconfirmed;
- withdrawn;
- replaced by a revised edition, or
- amended.

A bilingual version of this publication may be issued at a later date.

INTRODUCTION

Since the discovery of some Perovskite-type Cu-containing oxides, extensive research and development (R & D) work on high-temperature oxide superconductors has been, and is being, made worldwide, and its application to high-field magnet machines, low-loss power transmission, electronics and many other technologies is in progress.

In various fields of electronics, especially in telecommunication fields, microwave passive devices such as filters using oxide superconductors are being developed and are undergoing on-site testing [1,2]¹⁾.

Superconductor materials for microwave resonators, filters, antenna and delay lines have the advantage of very low loss characteristics. Knowledge of this parameter is of primary importance for the development of new materials on the supplier side and for the design of superconductor microwave components on the customer side. The parameters of superconductor materials needed for the design of microwave low loss components are the surface resistance R_s and the temperature dependence of the surface resistance.

Recent advances in high T_c superconductor (HTS) thin films with R_s several orders of magnitude lower than that of normal metals have increased the need for a reliable characterization technique to measure this property [3,4]. Traditionally, the R_s of Nb or any other low temperature superconducting material was measured by first fabricating an entire three dimensional resonant cavity and then measuring its Q -value. The R_s could be calculated by solving the EM field distribution inside the cavity. Another technique involves placing a small sample inside a larger cavity. This technique has many forms but usually involves the uncertainty introduced by extracting the loss contribution due to the HTS films from the experimentally measured total loss of the cavity.

The best HTS samples are epitaxial films grown on flat crystalline substrates and no high quality films have been grown on any curved surface so far. What is needed is a technique that: can use these small flat samples; requires no sample preparation; does not damage or change the film; is highly repeatable; has great sensitivity (down to 1/1 000th the R_s of copper); has great dynamic range (up to the R_s of copper); can reach high internal powers with only modest input powers; and has broad temperature coverage (4,2 K to 150 K).

The dielectric resonator method is selected among several methods [5,6,7] to determine the surface resistance at microwave frequencies because it is considered to be the most popular and practical at present. Especially, the sapphire resonator is an excellent tool for measuring the R_s of HTS materials [8,9].

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 provide an appropriate and agreeable technical base for the time being to 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.

1) Figures in square brackets refer to the Bibliography.

SUPERCONDUCTIVITY –

Part 7: Electronic characteristic measurements – Surface resistance of superconductors at microwave frequencies

1 Scope

This part of IEC 61788 describes measurement of the surface resistance of superconductors at microwave frequencies by the standard two-resonator method. The object of measurement is the temperature dependence of R_s at the resonant frequency.

The applicable measurement range of surface resistances for this method is as follows:

- Frequency: $8 \text{ GHz} < f < 30 \text{ GHz}$
- Measurement resolution: $0,01 \text{ m}\Omega$ at 10 GHz

The surface resistance data at the measured frequency, and that scaled to 10 GHz , assuming the f^2 rule for comparison, are reported.

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, *International Electrotechnical Vocabulary (IEV) – Part 815: Superconductivity*

3 Terms and definitions

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

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 dielectric resonator with the superconductor film specimen and then measuring the attenuation of the resonator at each frequency. The frequency shall be swept around the resonant frequency as the centre, and the attenuation–frequency characteristics shall be recorded to obtain Q -value, which corresponds to the loss.

The target precision of this method is a coefficient of variation (standard deviation divided by the average of the surface resistance determinations) that is less than 20 % for the 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 to determine the applicability of regulatory limitations prior to use.

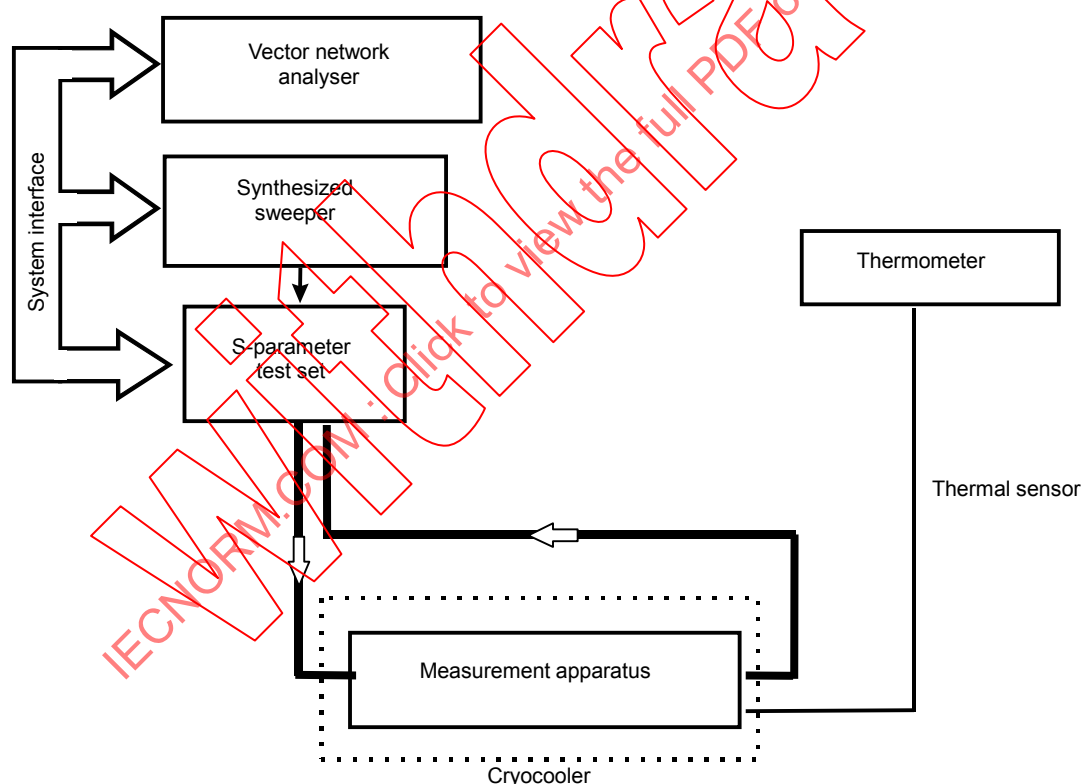
Hazards exist in this type of measurement. The use of a cryogenic system is essential to cool the superconductors to allow transition into the superconducting state. Direct contact of skin with cold apparatus components can cause immediate freezing, as can direct contact with a spilled cryogen. The use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

5 Apparatus

5.1 Measurement system

Figure 1 shows a schematic diagram of the system required for the microwave measurement. The system consists of a network analyzer system for transmission measurement, a measurement apparatus, and a thermometer for monitoring the measuring temperature.

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



IEC 004/02

Figure 1 – Schematic diagram of measurement system for temperature dependence of R_s using a cryocooler

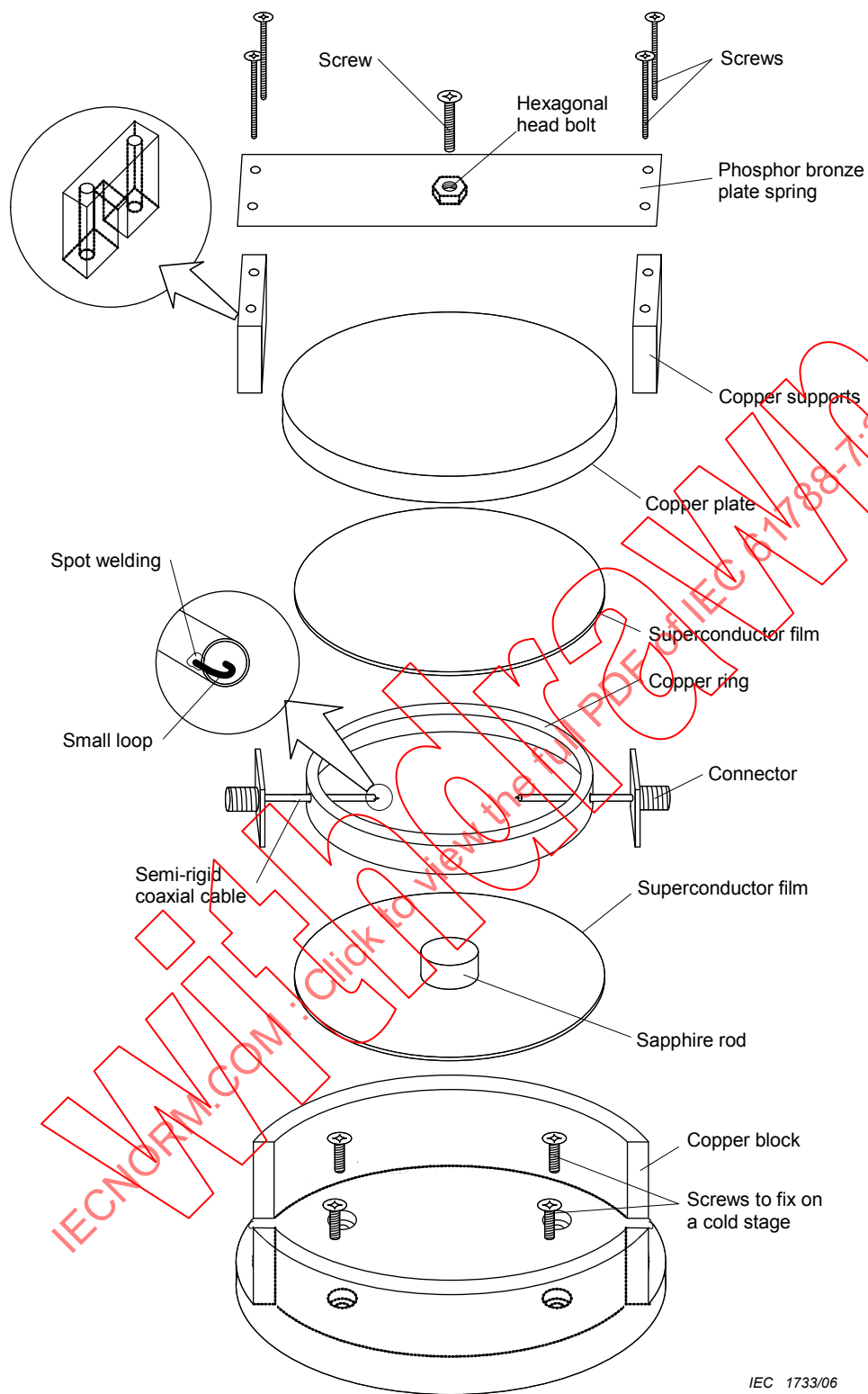
The measurement apparatus is fixed in a temperature-controlled cryocooler.

For the measurement of R_s for superconductor films, a vector network analyzer is recommended. A vector network analyzer has better measurement accuracy than a scalar network analyzer due to its wide dynamic range.

5.2 Measurement apparatus for R_s

Figure 2 shows a schematic of a typical measurement apparatus (closed type resonator) for the R_s of superconductor films deposited on a substrate with a flat surface. The upper superconductor film is pressed down by a spring, which is made of phosphor bronze. The plate type spring is recommended to be used for the improvement of measurement accuracy. This type of spring reduces the friction between the spring and the other part of the apparatus, and allows the smooth movement of superconductor films due to the thermal expansion of the dielectric rod. In order to minimize the measurement error, the sapphire rod and the copper ring shall be set in coaxial.

Two semi-rigid cables for measuring transmission characteristics of the resonator shall be attached on both sides of the resonator in an axial symmetrical position ($\phi = 0$ and π , where ϕ is the rotational angle around the central axis of the sapphire rod). Each of the two semi-rigid cables shall have a small loop at the ends. 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 cavity modes to the interested dielectric resonance mode shall be suppressed. Unwanted, parasitic coupling to the other modes reduces the high Q value of the TE mode resonator. For suppressing the parasitic coupling, special attention shall be paid to designing high Q resonators. Two other types of resonators along with the closed type shown in Figure 2 can be used. They are explained in Clause A.4.



IEC 1733/06

Figure 2 – Typical measurement apparatus for R_s

A reference line made of a semi-rigid cable shall be used to measure the full transmission power level, i.e., the reference level. This cable has a length equal to the sum of the two cables of the measurement apparatus. The semi-rigid cable with the outer diameter of 1,20 mm is recommended.

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

5.3 Dielectric rods

Two dielectric rods with the same relative permittivity, ϵ' , and loss factor, $\tan \delta$, preferably cut from one cylindrical dielectric rod, are required. These two rods, standard dielectric rods, shall have the same diameter but different heights: one has a height three times longer than the other.

It is preferable to use standard dielectric rods with low $\tan \delta$ to achieve the requisite measurement accuracy on R_s . Recommended dielectric rods are sapphire rods with $\tan \delta$ less than 10^{-6} at 77 K. Specifications on the sapphire rods are described in 7.1. In order 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 for the sapphire rods are described in Clause 7.

The diameter and the heights of the standard sapphire rods shall be carefully designed so that the TE_{011} and TE_{013} modes do not couple to other TM, HE and EH modes, since the coupling between TE mode and other modes causes the degradation of unloaded Q . A design guideline for the standard sapphire rods is described in Clause A.5. Table 1 shows typical examples of dimensions of the standard sapphire rods for 12 GHz, 18 GHz, and 22 GHz resonance. At higher frequencies the unloaded Q value will be lower, which makes the measurement easier, and the error will be lower.

Table 1 – Typical dimensions of pairs of standard sapphire rods for 12 GHz, 18 GHz and 22 GHz

Frequency GHz		Diameter	Height
		d mm	h mm
12	Short rod (TE_{011} resonator)	11,4	5,7
	Long rod (TE_{013} resonator)	11,4	17,1
18	Short rod (TE_{011} resonator)	7,6	3,8
	Long rod (TE_{013} resonator)	7,6	11,4
22	Short rod (TE_{011} resonator)	6,2	3,1
	Long rod (TE_{013} resonator)	6,2	9,3

6 Measurement procedure

6.1 Specimen preparation

From error estimation, the film diameter shall be about three times larger than that of the sapphire rods. In this configuration, the reduction in precision of R_s due to the different radiation losses between TE_{011} and TE_{013} mode can be considered negligible, given the target precision of 20 %. The film thickness shall be about three times larger than the London penetration depth value at each temperature. If the film thickness is much less than three times the London penetration depth, the measured R_s should mean the effective surface resistance.

Table 2 shows dimensions of the superconductor films recommended for the standard sapphire rods of 12 GHz, 18 GHz, and 22 GHz.

Table 2 – Dimensions of superconductor film for 12 GHz, 18 GHz, and 22 GHz

Standard dielectric rod		Superconductor film	
Frequency	Diameter	Diameter	Thickness
GHz	d mm	d' mm	μm
12	11,4	>35	$\approx 0,5$
18	7,6	>25	$\approx 0,5$
22	6,2	>20	$\approx 0,5$

In case of using closed type resonators, the dimensions of the superconductor films shall also be designed taking into account the dimension of the copper cylinder between the superconductor films. A design guideline for the dimension of the copper cylinder of the closed type resonator is described in Clause A.6.

6.2 Set-up

Set up the measurement equipment as shown in Figure 1. All of the measurement apparatus, standard sapphire rods, and superconductor films shall be kept in a clean and dry state as high humidity may degrade the unloaded Q -value. The specimen and the measurement apparatus shall be fixed in a temperature-controlled cryocooler. The specimen chamber shall be generally evacuated. The temperatures of the superconductor films and standard sapphire rods shall be measured by a diode thermometer, or a thermocouple. The temperatures of the upper and lower superconductor films, and standard sapphire rods must be kept as close as possible. This can be achieved by covering the measurement apparatus with aluminum foil, or filling the specimen chamber with helium gas.

6.3 Measurement of reference level

The level of full transmission power (reference level) shall be measured first. Fix the output power of the synthesized sweeper below 10 mW because the measurement accuracy depends on the measuring signal level. Connect the reference line of semi-rigid cable between the input and output connectors. Then, measure the transmission power level over the entire measurement frequency and temperature range. The reference level can change several decibels when temperature of the apparatus is changed from room temperature to the lowest measurement temperature. Therefore, the temperature dependence of the reference level must be taken into account.

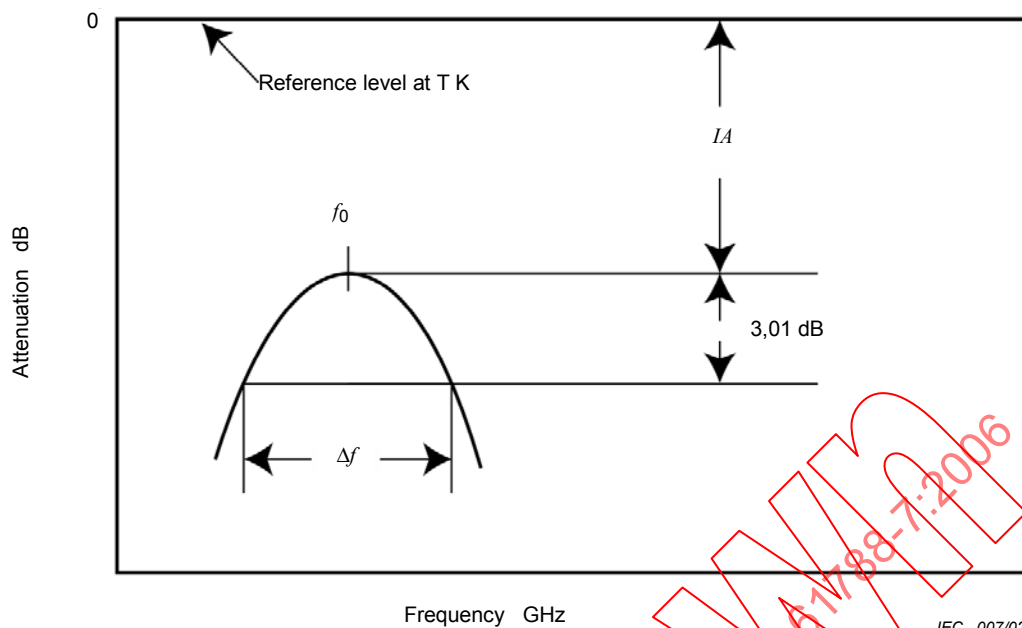


Figure 3 – Insertion attenuation IA , resonant frequency f_0 and half power bandwidth Δf , measured at T Kelvin

6.4 Measurement of the frequency response of resonators

The temperature dependence of the surface resistance R_s can be obtained through the measurements of resonant frequency f_0 and unloaded quality factor Q_u for TE_{011} and TE_{013} resonators, which shall be measured as follows.

- Connect the measurement apparatus between the input and output connectors (Figure 1). Insert the standard short sapphire rod near the centre of the lower superconductor film and fix the distance between the rod and each of the loops of the semi-rigid cables to be equal to each other, so that this transmission-type resonator can be under-coupled equally to both loops. Put down the upper superconductor film gently to touch the top face of the rod. Be careful not to damage the surface of the superconductor films by excessive pressure. Evacuate and cool down the specimen chamber below the critical temperature.
- Find the TE_{011} mode resonance peak of this resonator at a frequency nearly equal to the designed value of f_0 .
- Narrow the frequency span on the display so that only the resonance peak of TE_{011} mode can be shown (Figure 3). Confirm that the insertion attenuation IA of this mode is larger than 20 dB from the reference level, which depends strongly on the temperature.
- Measure the temperature dependence of f_0 and the half power band width Δf . The loaded Q , Q_L , of the TE_{011} resonator is given by

$$Q_L = \frac{f_0}{\Delta f} \quad (1)$$

- The unloaded Q -value, Q_u , shall be extracted from the Q_L by at least one of the two techniques described below.

One technique for extracting the unloaded Q -value involves measuring the insertion attenuation IA . The Q_u is given by

$$Q_u = \frac{Q_L}{1 - A_t}, \quad A_t = 10^{-IA[\text{dB}]/20} \quad (2)$$

This technique of using insertion attenuation assumes that the coupling on both sides of the resonator is identical. The coupling loops are difficult to fabricate, the orientation of the loop is difficult to control, and any movement of the sapphire rod during measurement is not known. These assembly dependent effects are also temperature dependent. This potential asymmetry in coupling can result in large errors in calculating the coupling factor if the coupling is strong ($IA < \sim 10$ dB). If the coupling is weak enough ($IA > 20$ dB), asymmetry in the coupling becomes less important.

Another technique for extracting the unloaded Q -value involves measuring the reflection scattering parameters at the resonant frequency of both sides of the resonator.

$$Q_u = Q_L(1 + \beta_1 + \beta_2) \quad (3)$$

$$\beta_1 = \frac{1 - |S_{11}|}{|S_{11}| + |S_{22}|} \quad (4)$$

$$\beta_2 = \frac{1 - |S_{22}|}{|S_{11}| + |S_{22}|} \quad (5)$$

In the above equations, S_{11} and S_{22} are the reflection scattering parameters as shown in Figure 4, and are measured in linear units of power, not relative dB. β_1 and β_2 are the coupling coefficients.

This technique of using the reflection scattering parameters has two advantages. It does not require the additional step of calibration of the reference level and it gives a measurement of the coupling values for both sides of the resonator. This also has two disadvantages. It only works for a narrow band resonance (which is fortunately the case) and is limited by the dynamic range of the network analyzer in measuring the reflection coefficients.

A combination of the two techniques is an excellent “double” check and is therefore recommended.

- f) The f_0 and Q_u measured for this short rod are denoted as f_{01} and Q_{u1} . By slowly changing the temperature of the cryocooler, the temperature dependence of f_{01} and Q_{u1} shall be measured.
- g) After the temperature dependence measurement of f_{01} and Q_{u1} is finished, the measurement apparatus shall be heated up to room temperature.
- h) Then, replace the TE_{011} resonator in the apparatus with the TE_{013} resonator at room temperature, cool down the apparatus to a temperature lower than the critical temperature, and measure the temperature dependence of f_0 and Q_u of its TE_{013} resonance mode, denoted as f_{03} and Q_{u3} , in a similar way as the TE_{011} resonator case. When the length of the sapphire rod of the TE_{013} resonator is precisely three times longer than that of the TE_{011} resonator, the f_{03} of the TE_{013} resonator must coincide with f_{01} of the TE_{011} resonator. If carefully designed, the difference between f_{01} and f_{03} is usually very small ($< \sim 0,25\%$). We can treat as $f_0 = f_{01} = f_{03}$ in the calculations of 6.5.

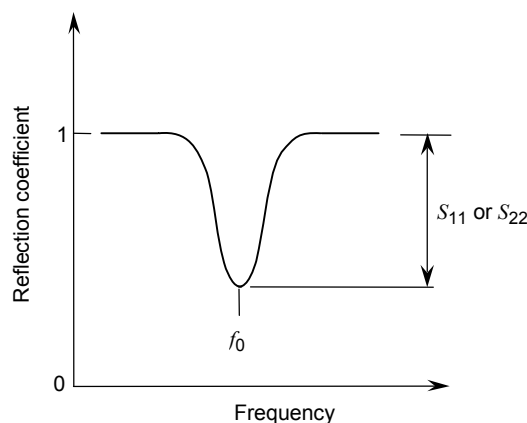


Figure 4 – Reflection scattering parameters (S_{11} and S_{22})

6.5 Determination of surface resistance of the superconductor and ϵ' and $\tan \delta$ of the standard sapphire rods

Calculate the temperature dependence of the surface resistance R_s of the superconductor films, and ϵ' and $\tan \delta$ of the standard sapphire rods using the temperature dependence of f_{01} , Q_{u1} , f_{03} , and Q_{u3} from Equations (6), (7) and (8).

$$R_s = \frac{30\pi^2 \times 3}{(3-1)} \left(\frac{2h_0}{\lambda_0} \right)^3 \frac{\epsilon' + W}{1+W} \left(\frac{1}{Q_{u1}} - \frac{1}{Q_{u3}} \right) \quad (6)$$

$$\epsilon' = \left(\frac{\lambda_0}{\pi d} \right)^2 (u^2 + v^2) + 1 \quad (7)$$

$$\tan \delta = \frac{1 + \frac{W}{\epsilon'}}{(3-1)} \left(\frac{3}{Q_{u3}} - \frac{1}{Q_{u1}} \right) \quad (8)$$

where

$$\lambda_0 = \frac{c}{f_0} \quad (9)$$

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

$$v^2 = \left(\frac{\pi d}{\lambda_0} \right)^2 \left[\left(\frac{\lambda_0}{2h_0} \right)^2 - 1 \right] \quad (11)$$

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

In the equations, λ_0 is the free space resonant wavelength, c is the velocity of light in a vacuum ($c = 2,9979 \times 10^8$ m/s), h_0 is the height of the short standard dielectric rod. 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. The derivations of the equations are described in Clause A.3.

Generally the thermal expansion coefficient of the rods must be known to determine the temperature dependence of their sizes. However, the thermal expansion effect of the sapphire rods can be neglected for the target precision of the R_s (20 %).

It is noted that the measured R_s means the effective surface resistance if the film thickness is not much larger than the temperature-dependent penetration depth.

7 Precision and accuracy of the test method

7.1 Surface resistance

The surface resistance shall be determined from the Q -value measured with a dielectric resonator technique.

A vector network analyzer as specified in Table 3 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 3 – Specifications for 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 dielectric resonators shall be provided with two dielectric rods with low $\tan \delta$ of less than 10^{-6} at 77 K and a radius less than 1/3 of the superconducting specimen's radius. The best candidate for the rods is sapphire as specified in Table 4. Term definitions in Table 4 are shown in Figure 5.

Table 4 – Specifications for sapphire rods

Diameter	$\pm 0,05$ mm
Height	$\pm 0,05$ mm
Flatness	below 0,005 mm
Surface roughness	top and bottom surface: below 10 nm r.m.s. cylindrical surface: below 0,001 mm r.m.s.
Perpendicularity	within 0,1 degree
Axis	parallel to c-axis within $0,3^\circ$

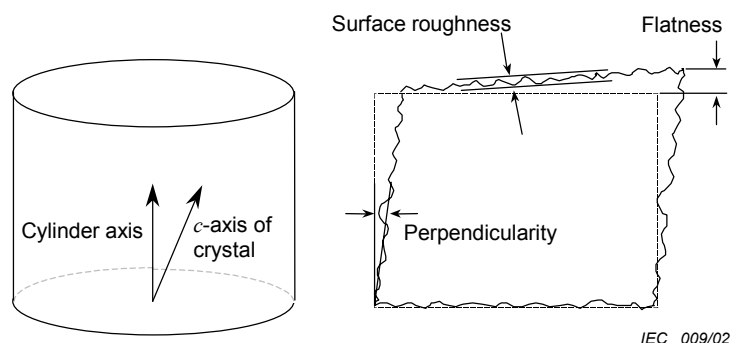


Figure 5 – Term definitions in Table 4

The technique as described assumes that single and triple height sapphire rods can be fabricated with the same $\tan \delta$. However, the variation of the $\tan \delta$ between nominally identical rods, cut from the same boule and polished by the same technique, may be as large as two orders of magnitude. To date, the smallest variation in $\tan \delta$ between nominally identical sapphire rods has been a factor of four[9]. Therefore, the uncertainty in the measured $\tan \delta$ is large. The variation of $\tan \delta$ of the present sapphire rod causes an additional uncertainty up to at least 10 % in the surface resistance measurement. This limits the target precision of the present technique at 20 %. If reproducibility of sapphire rods is improved, or a selection method for standard sapphire rods is established, a target precision can be improved.

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 dependence 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 the sample holder temperature. The holder temperature shall be reported to an accuracy of $\pm 0,5$ K, measured by means of an appropriate temperature sensor.

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

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 range of temperature.

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. A coating material thickness of less than several micrometres is recommended.

8 Test report

8.1 Identification of 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 composition of the thin film and the substrate;
- e) thickness and roughness of the thin film;
- f) manufacturing process technique.

8.2 Report of R_s values

The R_s values, together with their corresponding f_{01} , f_{03} , Q_{u1} , Q_{u3} , IA and/or (β_1, β_2) , ϵ' and $\tan \delta$ values, and their temperature 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 r.f. power;
- c) test temperature, accuracy of temperature and temperature difference in two plates;
- d) sample history with temperature variation.

Annex A (informative)

Additional information relating to Clauses 1 to 8

A.1 Scope

The establishment of the standard measurement method is needed to evaluate film quality of high T_c superconductor (HTS) films having low surface resistance R_s values such as 0,1 mΩ at 10 GHz. Several resonance methods as shown in Figure A.1 have been proposed so far to measure them in microwave and millimetre wave range. These resonator structures are grouped into the following six types:

A.1.1 Cylindrical cavity method [1]²⁾

Figure A.1(a) shows a cavity structure using the TE_{011} mode, which is constructed from a copper cylinder and two HTS films. In the microwave range, below 30 GHz, the R_s measurement precision of this method is rather low, because the R_s value of the copper cylinder is 100 times higher than that of HTS films. This method is suitable in the millimetre wave region, because the R_s values increase with f^2 of HTS films and $f^{1/2}$ for copper.

A.1.2 Parallel-plates resonator method [2]

Figure A.1(b) shows a structure of a parallel-plates resonator using the TM_{nm0} mode, which is constructed by inserting a thin low loss dielectric spacer between two rectangular HTS films. This method offers the possibility of measuring extremely small R_s values, but from the viewpoint of the absolute estimation of R_s there are some problems, such as low measurement precision of thickness and $\tan \delta$ of the dielectric spacer, uncertain estimations of radiation loss and an air-gap effect between the plates and the spacer, and critical excitation technique of the resonance modes.

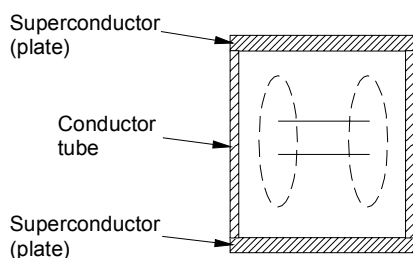
A.1.3 Microstrip-line resonance method [1], [3]

Figure A.1(c) shows a structure of a microstrip-line resonator using the TEM_n mode, which is fabricated by a pattern fabrication process and is close to the real microwave passive devices. However this method is not suitable for the HTS film estimation because it includes an uncertain influence of the pattern fabrication process.

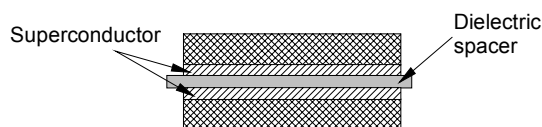
A.1.4 Dielectric resonator method [4], [5]-[7]

Figure A.1(d) shows a structure of a dielectric resonator using a TE_{011} mode, where a low loss sapphire rod is placed between two parallel HTS films. For the TE_{0mp} mode, we can eliminate the air-gap effects, because the normal component of electric field does not exist on the HTS films. In this method the R_s values of the HTS films have been measured by ignoring the effect of $\tan \delta$ on the assumption of $\tan \delta < 1 \times 10^{-8}$ for single-crystal sapphire [4]. However the $\tan \delta$ values measured for sapphire at 10 GHz and near 50 K, as is well known, take values between 10^{-6} to 10^{-8} due to quantity of lattice defect. Therefore preparation of very-low-loss sapphire rods with $\tan \delta < 1 \times 10^{-8}$ is essential for this method.

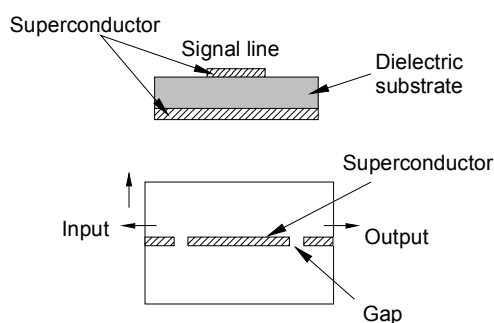
²⁾ Numbers in brackets in this annex refer to the reference documents in Clause A.9



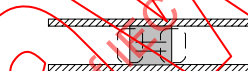
a) Cylindrical cavity resonator method



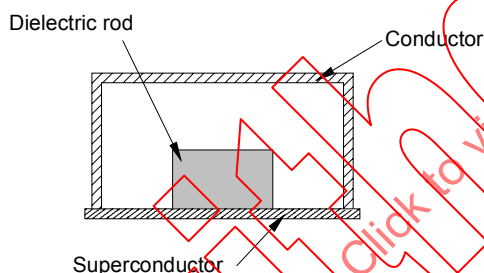
b) Parallel plate resonator method



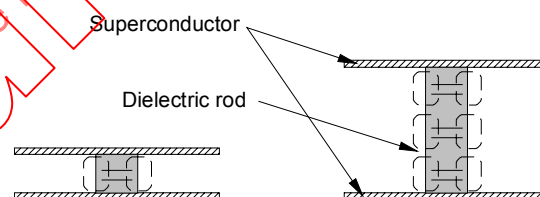
c) Microstrip resonator method



d) Dielectric resonator method



e) Image type dielectric resonator method



f) Two resonator method

— Electric field
- - - Magnetic field

IEC 010/02

Figure A.1 – Schematic configuration of several measurement methods for the surface resistance

A.1.5 Image-type dielectric resonator method [8]

Figure A.1(e) shows a structure of an image-type dielectric resonator using the $TE_{01\delta}$ mode. This structure is available to measure only one HTS film. However preparation of very-low-loss sapphire rods is essential also for this method, because the R_s is measured by ignoring the effect of $\tan \delta$.

In addition, complicated and tedious numerical calculations are needed to determine the R_s values from the measured resonant frequency and unloaded Q .

A.1.6 Two-resonator method [9], [10].

In this method two sapphire rod resonators with the same $\tan \delta$ values are used; one is a TE_{011} mode resonator and the other is a TE_{013} mode resonator, as shown in Figure A.1 f). Similarly to the dielectric resonator method, the air-gap effects can be eliminated in this method. The $\tan \delta$ value of the sapphire rods and the R_s value of the HTS films can be determined separately from the resonant frequencies and unloaded Q 's measured for these resonators by using simple formulas derived from the rigorous analysis based on the mode matching method. Thus this method can eliminate the ambiguity of the $\tan \delta$ affected to the R_s measurements. Actually, the results of R_s measured for six YBCO films with $R_s = 0,1 \text{ m}\Omega$ at 12 GHz have verified that the measurement precision of 10 % is realized by this method [10]. However, if there is a difference between two $\tan \delta$ values ($\Delta \tan \delta$), the errors of R_s due to $\Delta \tan \delta$ must be taken into account. This effect will be estimated from results of the inter-laboratory round robin test of the same HTS films by this method.

As a result of comparisons among the six methods described above, the two-resonator method is recommended as the standard measurement method of R_s of HTS films, due to the following two advantages:

- absolute measurement of R_s is possible;
- the numerical treatment is simple and easy.

A.2 Requirements

Higher measurement frequencies are desirable for determination of lower R_s because R_s of superconductor films increases with the frequency according to the f^2 rule. The size of the resonators also can be made smaller by using higher measurement frequencies. However, it becomes difficult to set up a microwave measurement system as the measurement frequency becomes higher.

For the measurement temperatures of below 30 K or above 80 K, this test method is sufficiently applicable, where some new cooling techniques may be required.

A.3 Theory and calculation equations

Figure A.2 shows the configuration of the TE_{0mp} mode resonator, which is used to eliminate the air-gap effects. A cylindrical dielectric rod with diameter, d , and height, h , is short-circuited at both ends by surfaces of two parallel superconductor films deposited on dielectric substrates with diameter, d' , thus constituting a resonator. These superconductor films are required to have the same value of R_s . The value of R_s is calculated from the measured resonant frequency f_0 and unloaded quality factor Q_u for the TE_{0mp} resonance mode. When the two superconductor films have different values of R_s , the measured R_s value corresponds to the average value of these two films.

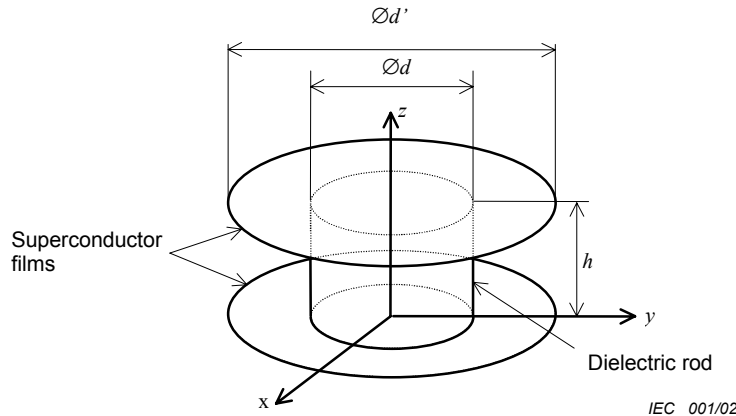


Figure A.2 – Configuration of a cylindrical dielectric rod resonator short-circuited at both ends by two parallel superconductor films deposited on dielectric substrates

The value of R_s is given by

$$R_s = \frac{1}{B} \left(\frac{A}{Q} - \tan \delta \right) \quad (\text{A.1})$$

where

$$A = 1 + \frac{W}{\varepsilon'} \quad (\text{A.2})$$

$$B = p^2 \left(\frac{\lambda_0}{2h} \right)^3 \frac{1+W}{30\pi^2 \varepsilon'}, \quad p = 1, 2, \dots, \quad (\text{A.3})$$

$$\lambda_0 = \frac{c}{f_0} \quad (\text{A.4})$$

$$W = \frac{J_1^2(u)}{K_1^2(v)} \frac{K_0(v)K_2(v) - K_1^2(v)}{J_1^2(u) - J_0(u)J_2(u)} \quad (\text{A.5})$$

$$v^2 = \left(\frac{\pi d}{\lambda_0} \right)^2 \left[\left(\frac{p \lambda_0}{2h} \right)^2 - 1 \right] \quad (\text{A.6})$$

$$u \frac{J_0(u)}{J_1(u)} = -v \frac{K_0(v)}{K_1(v)} \quad (\text{A.7})$$

In Equations (A.1) and (A.2), ε' and $\tan \delta$ are the relative permittivity and the loss factor of the dielectric rod, respectively. In Equations (A.3) and (A.4), λ_0 is the free space resonant wavelength, and c is the velocity of light in a vacuum ($c = 2,9979 \times 10^8$ m/s). The function W/ε' equals the ratio of electric-field energy stored outside to that stored inside the dielectric rod. If all of the electric field is concentrated inside the dielectric rod, the value W equals zero.

The value u^2 is given by the transcendental Equation (A.7) 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 $J_1(u_{1m}) = 0$. The first solution ($m = 1$), which is used for easy mode identification, is shown in Figure A.3 by curve (A). The computed result of the W - v relation for $m = 1$ of TE_{0mp} resonance mode is shown in Figure A.3 by curve (B).

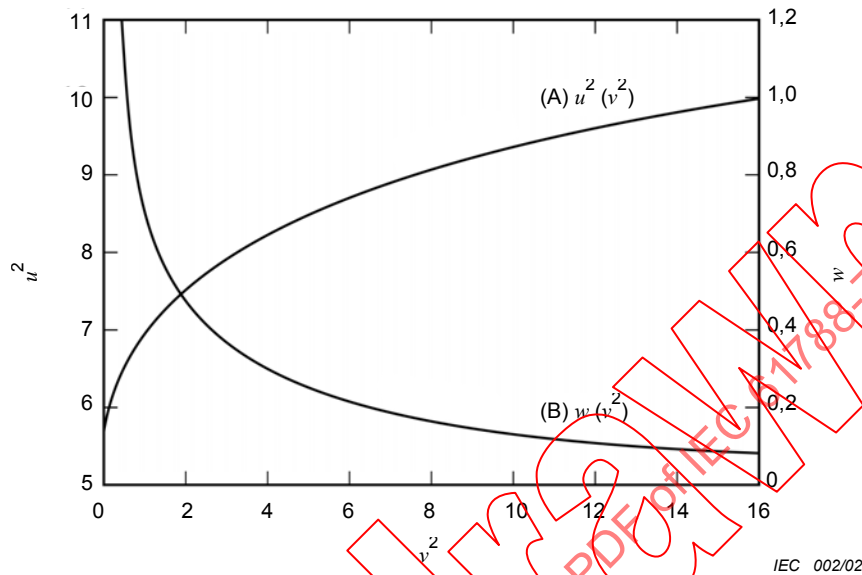


Figure A.3 – Computed results of the u - v and W - v relations for TE_{01p} mode

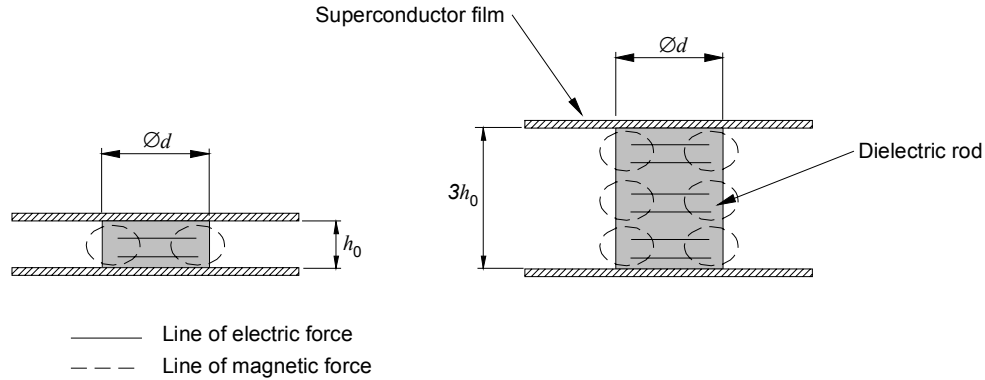
The value of ε' is given by

$$\varepsilon' = \left(\frac{\lambda_0}{\pi d} \right)^2 (u^2 + v^2) + 1 \quad (\text{A.8})$$

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

In the two-resonator method, a pair of dielectric rods, which are called "standard dielectric rods", are used. These two rods have the same diameter but have different heights. The rod heights are such that one rod is p times the height of the other; p is commonly set equal to three. They are required to have the same values of ε' and $\tan \delta$.

Figure A.4 shows the configuration of the standard dielectric rods in the case of $p = 3$. To avoid confusion, the height of the short standard dielectric rod is denoted by h_0 . Each resonator is called " TE_{011} resonator" and " TE_{013} resonator", respectively. The same superconductor films are used in these resonators. The values of f_0 and Q_u for the TE_{011} mode are measured using the TE_{011} resonator, and those for the TE_{01p} mode are measured using the TE_{01p} resonator. We denote the f_0 and Q_u for each resonator by using the subscripts 1 and p , respectively: f_{01} and Q_{u1} for TE_{011} resonator, and f_{0p} and Q_{up} for TE_{01p} resonator.



IEC 003/02

Figure A.4 – Configuration of standard dielectric rods for measurement of R_s and $\tan \delta$

The value of $\tan \delta$ is given from the measured values of Q_u . When the TE_{01p} resonator is precisely p times longer than the TE_{011} resonator, f_{0p} coincides with f_{01} . However Q_{up} is higher than Q_{u1} according to the different magnitude of the electric field energy stored in the two resonators. Owing to the fact that both dielectric rods are short-circuited at both ends by the same superconductor films, Equation (A.1) yields

$$\tan \delta = \frac{A}{(p-1)} \left(\frac{p}{Q_{up}} - \frac{1}{Q_{u1}} \right) \quad (A.9)$$

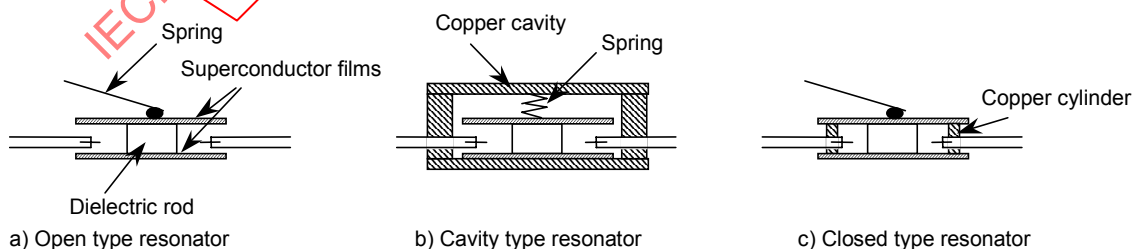
As an alternative method, the value of R_s of superconductor films can be directly measured by

$$R_s = \frac{30\pi^2 p}{(p-1)} \left(\frac{2k_0}{\lambda_0} \right)^3 \frac{\epsilon' + W}{1 + W} \left(\frac{1}{Q_{u1}} - \frac{1}{Q_{up}} \right) \quad (A.10)$$

where Equation (A.10) is derived by substituting Equation (A.9) into Equation (A.1).

A.4 Apparatus

Unwanted parasitic coupling to the other mode reduces the high Q -value of the TE mode resonator. For suppressing the parasitic coupling, special attention is paid to design high Q resonators. Three types of resonators are proposed and shown in Figure A.5:



IEC 011/02

Figure A.5 – Three types of dielectric resonators

- a) Open type resonator: a low loss dielectric 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 propagate along the coupling cables, which degrades the high Q of the TE_{0mp} mode and causes the increase in error for the R_s measurements. The coupling cables should be set as close as possible to the lower superconductor film.
- b) Cavity type resonator: the open type resonator shown in a) is placed in a conductor (copper) cavity.
- c) Closed type resonator: 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 vertical position of the coupling cables (z) at the highest z -axis magnetic field component (Hz), i.e. $z = h/2$, is recommended.

The measuring apparatus on the cryocooler is protected from mechanical and thermal disturbances, and installed in an X-Y and/or Z-axial manipulator for adjusting sample positions in the range of approximately ± 1 mm.

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

A.5 Dimensions of the standard sapphire rods

Figures A.6 and A.7 show the mode charts for designing the TE_{011} and TE_{013} mode resonators, respectively, short-circuited at both ends by parallel superconductor films, in which an uniaxial-anisotropic characteristics of the relative permittivity of the sapphire rod is taken into consideration [11]. ϵ_z is the relative permittivity in the direction of the c -axis, ϵ_r is that in the plane perpendicular to the c -axis, d is the diameter of the sapphire rod, and h is the height of the sapphire rod, and λ_0 is the free space resonant wavelength. As shown in figures A.6 and A.7, TE_{013} resonance mode is apt to be affected by TM or HE mode in comparison with TE_{011} resonance mode. Since the coupling between TE mode and other modes causes the degradation of unloaded Q , the (d/h) of the sapphire rods shall be selected so as to avoid the unwanted couplings.

As shown in Figure A.7, the value $(d/h)^2$ for the TE_{013} resonator is selected from 0,24 to 0,46 in order that the TE_{013} resonance mode is not affected by the other modes. This value $(d/h)^2 = 0,24$ to 0,46 leads to a value of $(d/h)^2 = 2,2$ to 4,1 for the TE_{011} resonator. The TE_{011} resonance mode does not also couple with other modes when the value $(d/h)^2$ is in the region of 2,2 to 4,1 as shown in Figure A.6.

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

From the curve of TE_{011} mode in Figure A.6, the value of $\epsilon_r (d/\lambda_0)^2$ can be determined for each $(d/h)^2$ value. When the value $(d/h)^2$ equals 4,0, for example, the value of $\epsilon_r (d/\lambda_0)^2$ equals 1,92. Thus, the resonant frequency of TE_{011} mode for the sapphire rod with dimension of $(d/h)^2 = 4,0$ is calculated from the following equation by specifying d and ϵ_r of the sapphire rod:

$$\epsilon_r (d/\lambda_0)^2 = \epsilon_r (d \times f_0 / c)^2 = 1,92 \quad (\text{A.11})$$

A.6 Dimension of the closed type resonator

In the closed type resonator, a copper cylinder is put between the superconductor films and a sapphire rod is placed in the center of the copper cylinder (Fig. A.5(c)). The resonant frequency of each mode changes with the inner diameter of the copper cylinder, D . Therefore D shall be selected so as to avoid the unwanted coupling with the other modes. In Figures A.8 and A.9, mode charts of the closed type resonators are shown for the short sapphire rod ($(d/h)^2 = 4$) and the long sapphire rod ($(d/h)^2 = 0,44$), respectively, as a function of $S = D/d$ [11]. The ranges of S to separate the TE_{011} and the TE_{013} modes simultaneously from the others are $S = 1,8 \sim 2,8$, $3,8 \sim 4,1$ and $4,8 \sim 5,2$. The value of $S = D/d = 4$ is recommended because of the ease with which it can be treated.

A.7 Precision and accuracy of the test method

Errors estimation [4][10], sensitivity, accuracy and repeatability for these methods can be obtained by the error analysis concerning R_s as well as by the round robin test.

A.8 Sapphire rod reproducibility

Knowing the value of $\tan \delta$ of the sapphire rod is a difficult problem. One approach based on the single and triple height rods involves the selection of a standard rod for each type. The first step is to compare a large number of sapphire rods by measuring them in a single HTS/sapphire resonator. The sapphire rod with the highest unloaded Q -value is the temporary label of standard. This procedure is repeated for a set of triple height rods. The “standard” single and triple height rods are then used as described above to obtain $\tan \delta$. The electrical properties, ε' and $\tan \delta$, of other rods can then be calibrated by a direct comparison with these standard rods. Using a calibrated single height rod it is then possible to extract the R_s values of superconductor films under test.